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The New York section of the American Chemical Society will be happy to contribute by all possible means to the comfort and enjoyment of the foreign chemists who may visit New York on their way to or from Chicago during the World's Fair.

Visitors are requested to leave their names and addresses with the Secretary of the Reception Committee, who will also be glad to furnish all information at his command in reply to letters of inquiry.

For the Reception Committee,

H. CARRINGTON BOLTON.

MORRIS LOEB, Secretary,
University of the City of New York,
Washington Square, East,
room 16.

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THE JOURNAL
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[CONTRIBUTIONS FROM CHEMICAL LABORATORY, U. S. DEPARTMENT OF AGRICULTURE.—NO. 4.]

LAMP FOR CONSTANT MONOCHROMATIC FLAME.[1]

BY H. W. WILEY.

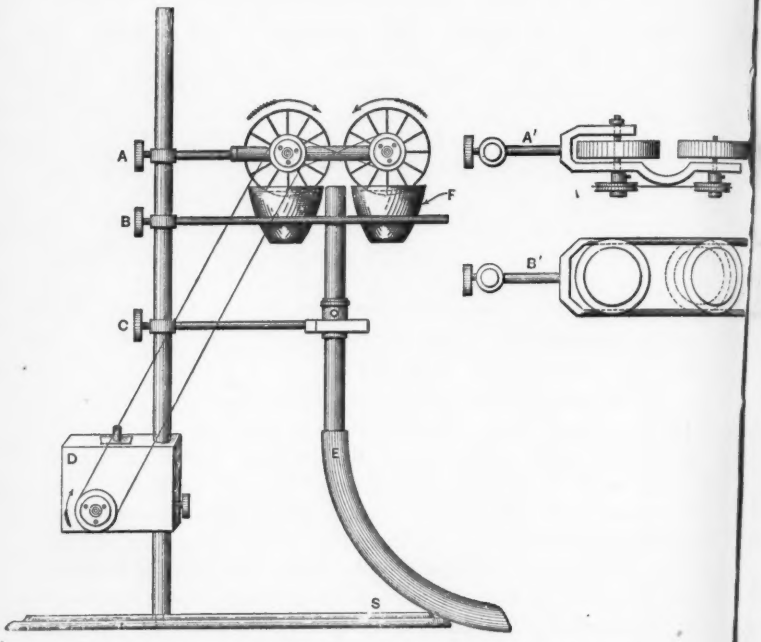
Received May 3, 1893.

IN optical experiments requiring a constant monochromatic flame there is great difficulty in securing an intensity of illumination which remains constant for an indefinite time. The ordinary lamps, for instance, producing a sodium flame, are fed by introducing, from time to time, fragments of fused salt into a platinum mesh spoon which touches the edge of the flame. The intensity of the illumination at the time of adding a fresh portion of the fused salt is great, but after a few minutes there is a sensible difference. For this reason, polarimetric observations are rendered somewhat difficult, inasmuch as the instrument which is set at zero with one intensity of illumination would vary somewhat from that with a different illumination. Probably this would not be the case if absolutely pure chemicals could be used. The only difference then would be in the intensity of the light. In point of fact, however, a difference in the color of the light is often disclosed with the lamps of ordinary construction, so that an instrument which is set at zero at the moment a fresh piece of fused salt is placed in the flame would not show the same zero after the salt had been ignited for some time.

To avoid such difficulties as these and secure a constant,

[1] Presented to Washington Chemical Society, April 13, 1893.

uniform coloration for polarimetric observations, the lamp which is to be described has been devised.



It consists essentially of two wheels with platinum gauze perimeters and platinum wire spokes, driven by a clock-work, D, and mounted by the supports A A', as shown in the figure. The sodium salt, chlorid or bromid, in saturated solution, is placed in the porcelain crucibles, F, supported by B B', as indicated in the figure, to such a depth that the rims of the platinum wheels dip beneath the surface as they revolve. The salt is volatilized by the lamp, E. By means of the crossed bands the wheels are made to revolve in opposite directions as indicated by the arrows. The solution of the salt, which is taken up by the platinum net work of the rim of the wheel, thus has time to become perfectly dry before it enters the flame, and the sputtering which a moist salt would produce is avoided.

At every instant, by this arrangement, a minute fresh portion of salt is introduced into the flame with the result of making a perfectly uniform light, which can be used for hours without any perceptible variation. The mechanism of the apparatus is so simple that no further description is necessary. The polariscope should be so directed toward the flame as to bring into the field of vision its most luminous part. The platinum wheels are adjustable and should be so arranged as to produce between them an unbroken yellow flame. The wheels are eight cm. in diameter and driven at a rate to make one revolution in six to ten minutes.

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY U. S. DEPARTMENT OF AGRICULTURE, NO. 5.]

IMPROVED EXTRACTION APPARATUS.

BY H. W. WILEY.

Received May 26, 1893.

THE apparatus for the extraction of substances soluble in ether, alcohol, etc., described in the *Journal of Analytical and Applied Chemistry* for February, 1893, pp. 65, *et seq.*, can be more conveniently operated when constructed in the manner to be described.

It is convenient to have the bath for holding the tubes made in two separate portions, K and K′. The box K can be conveniently made of galvanized iron with legs, U and U′, of any convenient length, so that a lamp can be placed underneath the box.

The liquid to be used in the bath may be water or other substance of different boiling point, and should stand at the height represented by the line W. The box has a false bottom represented by the dotted line O, with circular perforations to receive the bottom of the extraction tubes, as indicated. Both sides of this box are conveniently made of glass or mica so that the operator can see the progress of the evaporation of the solvent.

This box K′ is to rest lightly on K but is not fastened to it in any way. It is also conveniently made with one or both sides of glass or mica. The bottom of the box carries rubber diaphragms, perforated to receive the extraction tubes, through

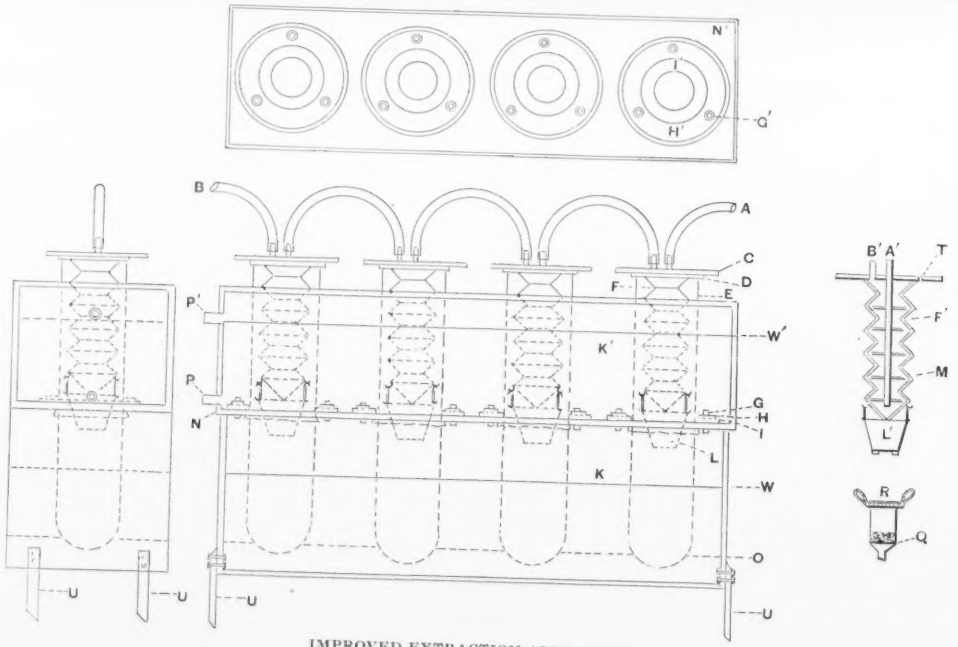
which the tubes pass water-tight. The plan of the bottom is shown in figure H′ N′ G′. The rubber diaphragms are held by two brass rings fastened together by small bolts. One of these rings is placed below the perforated bottom and one above. The perforation of the rubber diaphragms is shown at I′. An end view of the box is also shown at the left of the illustration.

This box has two exits, P and P′, so that water can be allowed to flow in a thin film over the bottom and escape at the exit P, or if the exit P be closed the water can be made to rise to the level of P′ as shown by the line W′. The outside of the test tubes can in this way be surrounded with water, or if a lower temperature be required fragments of ice can be dropped in. The object of this is to condense the vapors of the solvent which may strike against the sides of the tube and thus tend to rise to the top and escape at the intersection of the metal plate and the ground glass rim of the tube.

The baths are made of convenient size to hold four of these tubes and the condensers can all be cooled with a single stream of water entering at A and after passing through the four condensers escaping at B. A section of the condenser is shown at the right of the illustration. The water enters through A′ and passing upward round the disks M is thrown to the outside of the condenser and escapes at B′. The vent at T is left open until the vapor of the solvent has filled the interior of the apparatus, when it is closed.

The material to be extracted may be held in the perforated crucible L′ or an ordinary glass extraction tube, R, Q, with a perforated platinum disk fused into the glass at Q, and carrying an asbestos felt, can be employed. The upper part of the glass extraction tube has a little flange on it to support a looped wire, by means of which it can be hung to the hooks on the condenser in the same manner that the crucible is supported. The advantage of using glass is that the operator can see every thing which is going on in the extracting tube.

When the apparatus is to be used with ether the box K′ should be lifted up from K and the extraction tubes thrust farther through the diaphragm, until at least one of the corrugations of the condenser passes below the diaphragm. Otherwise



IMPROVED EXTRACTION APPARATUS.

the ether will be largely condensed on the sides of the glass test tubes and will run back down the sides instead of being collected on the condenser and dropped into the extraction tube. The box K′, for this purpose, can be held on any convenient support. When ether is used the vent P is closed and the one at P′ left open and a stream of water is made to flow into the box at the end opposite P′ so that the water in K′ is continually changing. This avoids any objectionable loss of ether by its escaping at the top of the apparatus.

When eighty per cent. alcohol is used for the extraction the vent at P is to be left open and sufficient water allowed to run in at the end opposite P to keep the bottom of the box K′ cool. The vapor rising from the box K is condensed by this cooled surface so that it drops directly back into K. When alcohol is used the box K′ should rest directly upon K.

With eighty per cent. alcohol the boiling point of water is not quite sufficient to secure a rapid evaporation of the solvent. A mixture composed of two parts of glycerol and one part of water will be found most convenient for this purpose. Inasmuch as many of the substances extracted by alcohol give up a large quantity of matter to the solvent the boiling point is therefore considerably raised and the glycerol bath is necessary in this case to secure a sufficiently rapid evaporation. To avoid any danger of bumping with such alcoholic solutions, it is convenient to add a few fragments of platinum foil.

The residues in the test tubes can be dried and weighed in the tubes or the amount of the extracted matter can be determined by weighing the crucibles or glass tubes containing the matter to be extracted. The latter method is preferable, with perfectly dry substances inasmuch as it is possible especially with ether that some bodies may be extracted from the material which are subsequently volatilized during the drying of the extract. Whenever it is desired to weigh the extract, especially the ether extract, it may be received in a small cylindrical glass vessel with flat bottom, four to six cm high and of a diameter just permitting its introduction into the extraction tube. About ten cc. of mercury are first poured into the extraction tube on which this vessel rests. The necessity of weighing the heavy

test tube is thus avoided and the extract is more easily dried in the small vessel.

This construction of the apparatus is compact, occupying but a small space; is easily manipulated, requiring but little attention, and is economical in respect of the use of gas, a single lamp being sufficient to operate a bath containing four tubes. It has the additional advantage that all parts are open to inspection so that the progress of the extraction can be watched at all times if necessary. It also has the advantage that both the extract and the residue can be weighed and thus a check on the results be obtained. The volatilization of the solvent should be so regulated as not to accumulate any liquid in the extracting tube.

ABSOLUTE ALCOHOL.

SECOND PAPER.

By Edward R. Squibb, M. D., of Brooklyn, N. Y.

Read before the New York Section, June 2, 1893.

A VERY considerable experience in the manufacture of so-called "absolute alcohol" for the market up to 1884 convinced the writer that really anhydrous alcohol had not yet been obtained.

During the latter part of 1883 and the early part of 1884 the subject was investigated with care, and the results were published by the writer in the *Ephemeris* for May, 1884, **2**, 522. This paper opens with the following paragraphs, which are as true to-day as they were ten years ago:

"It appears to be very certain that no alcohol has as yet been rendered entirely anhydrous, and therefore the term 'absolute,' as applied to any yet made, is not strictly correct. For all practical purposes, however, it is a very convenient term by which to designate a rather indefinite substance, but one now applied to a great many important uses, and therefore itself growing in importance.

"It is not difficult to get alcohol practically free from all impurities, including water as one, but to free it from the last one-thousandth part of water is very difficult indeed—so difficult, that traces of this ultimate fraction of water have, so far, always been retained. Hence in considering it as absolute alcohol, it can only be regarded as being freer from all other impurities than from water, and as being more or less free from

water. No alcohol should be called absolute, however, that contains less than 99.4 per cent. by the best determinations. When carefully freed from all impurities except water, specific gravity becomes a very accurate indication of strength, and it has always been relied upon as the decisive test of strength. For many years each careful observer reduced the specific gravity little by little to those specific gravities upon which the tables of the present day are based, by using new appliances which the progress of knowledge supplied, and the object of this note is to show that improvement in these appliances is not yet at an end."

The paper then goes on to show that by slow, cold percolation through quick-lime and simple distillation, alcohol in large quantities had been obtained that was of lower specific gravity than the lowest given by the best of the authorities on the subject. Over thirty determinations were found, and hardly any two of these were in fair accord. Some of the best and most generally quoted of these authorities are examined and discussed with the conclusion that it is particularly unfortunate that the three most generally accepted authorities should, by the construction of their elaborate tables upon erroneous bases, have misled the world for so many years upon this enormous alcohol interest.

Then, commencing with an alcohol that was already below all the authorities found, and yet known to contain traces of water, the attempt was made to reach a still lower degree of hydration. The means adopted, and the checks upon the work, and the results are carefully described in detail with the hope and expectation that more capable hands would go over the work to correct or confirm it, since it was much too important to be accepted without confirmation by other investigators. Nearly nine years have now passed since the publication of this paper, and yet notwithstanding the importance of the subject, no further investigations have been noticed.

The specific gravities previously reached were, at $\frac{15.6^\circ}{15.6^\circ}$ C., 0.79381 (Drinkwater);[1] $\frac{15.6^\circ}{15.6^\circ}$ C., 0.7938 (Fownes);[2] $\frac{15.6^\circ}{15.6^\circ}$ C., 0.7939 (Tralles);[3] $\frac{15^\circ}{4^\circ}$ C., 0.79367 (Mendelejeff).[4] This last specific gravity when brought to the terms of the others, namely $\frac{15.6^\circ}{15.6^\circ}$ C., is 0.79391.

[1] Quoted from *Memoirs of the Chemical Society*, **3**, 450.
[2] Quoted from *Fownes' Manual of Chemistry*, third edition, 591.
[3] Quoted from *Watt's Dictionary of Chemistry*, *1888*, **1**, 95.
[4] Quoted from Roscoe and Schorlemmer, *Treatise on Chemistry*, **3**, Part I, 299.

The specific gravity reached at that investigation was, at $\frac{4^\circ}{4^\circ}$ C., 0.802566; at $\frac{15^\circ}{4^\circ}$ C., 0.793260; at $\frac{15.6^\circ}{4^\circ}$ C., 0.79279; at $\frac{25^\circ}{4^\circ}$ C., 0.78496; at $\frac{15.6^\circ}{15.6^\circ}$ C., 0.79350; at $\frac{25^\circ}{15.6^\circ}$ C., 0.78573. Thus the reduction of specific gravity effected by the work of that paper was from about 0.79380 to 0.79350 at the temperature of 15.6 C. (60° F.) compared with water at the same temperature of 15.6 (60° F.), or 0.00030, which in strength is equivalent to nearly one-tenth of one per cent.—0.000338 being the difference in specific gravity for 0.10 per cent.

Judging from the circumstances attending the percolations and distillations and from the want of constancy in the boiling point as shown by slight differences in specific gravity of different fractions of distillate, the writer was convinced that this alcohol was not quite anhydrous, and supposed that by modification of process and by better management a specific gravity of about 0.79340 should be attained. The further experience of the past nine years has rather tended to confirm that judgment, and therefore it was concluded to return to the subject at this time, and to review the former work with different methods and some modifications of process, and some improvements in apparatus suggested by longer experience.

APPARATUS OF PRECISION FOR THE SPECIFIC GRAVITY OF LIQUIDS.

As specific gravity is the sole indication of strength to be relied on, and as alcohol changes very much in volume by changes in temperature, accurate and highly sensitive apparatus was of the first importance in the determination, and it was thought that improvements could be made in the apparatus formerly used.

A standard thermometer was used, made by Hicks of London from Powell glass, standardized and graduated at Kew in 1884, about eight years after it was made. This instrument had its present error determined by comparison with the international standards at the U. S. Bureau of Weights and Measures in Washington through the kindness of the superintendent, Prof. T. C. Mendenhall, and its indications of temperature were read to 0.1° C.

Several very accurate specific gravity bottles of 100 and 500

AMERICAN CHEMICAL SOCIETY.

FIFTH GENERAL MEETING.

CHICAGO, AUGUST 21, 1893.

THE following resolution was adopted by the Council of the American Chemical Society, June 7, 1893:

Resolved, That the General Meeting of the American Chemical Society for the summer of 1893 be held in Chicago at such date as may be determined by the committee appointed by the society to co-operate with the World's Fair Auxiliary of the Columbian Exposition in arranging for an International Congress of Chemists.

In accordance with the terms of this resolution the committee to co-operate with the World's Fair Auxiliary have determined that the Fifth General Meeting of the Society shall be held during a period beginning August 21, 1893, in conjunction with the World's Congress of Chemists to be held under the auspices of the World's Fair Auxiliary and the American Chemical Society.

The committee desire to call to the attention of the members of the society and of American chemists generally, the fact that in view of the large number of eminent chemists, home and foreign, who have already signified an intention to attend the World's Congress of Chemists, and the valuable character of the papers that will be presented, this meeting will doubtless be the most important gathering ever held by chemists in the United States. The time and place are therefore particularly auspicious, and the promise of a most successful and enjoyable

occasion very great. The meetings will be open to all chemists in good standing, and in order that a program of proceedings may be arranged in advance, it is earnestly requested that those who desire to offer papers will forward titles thereof as early as August 1, to Prof. Wm. McMurtrie, 106 Wall street, New York City, care of New York Tartar Co.

It is also the hope of the committee that in view of the important part taken by the American Chemical Society in calling the World's Congress of Chemists, the society will be represented by a large number of its members in this meeting. This is due to the foreign chemists who have accepted the invitations extended by the society.

In matters of transportation to, and quarters in, Chicago, members of the society will have all the advantages provided for those who will attend the World's Congress of Chemists. Those desiring information with this regard may obtain it by addressing Prof. John H. Long, No. 2421 Dearborn avenue, Chicago, Ill.

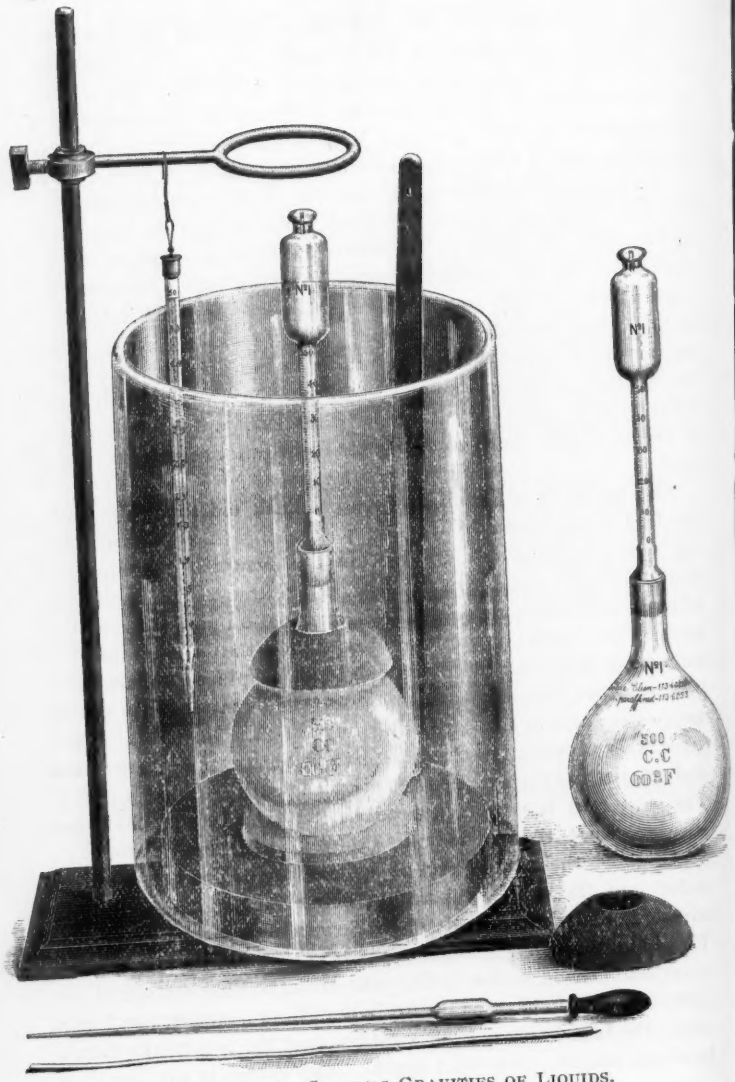
It is requested that as soon as possible after their arrival in Chicago, members will report at the Congress Headquarters in the Art Institute Building, Lake Front and Adams street, and register their names and Chicago addresses so that they may receive without delay such notices and information as the committees may have to distribute at that time.

WM. McMURTRIE,
C. F. CHANDLER,
A. B. PRESCOTT,
C. E. MUNROE,
H. W. WILEY,

Committee to Co-operate with the World's Fair Auxiliary.

grams were used, but the one relied upon for the final determinations, and shown in the accompanying cut, is supposed to be an improvement upon any hitherto described. The device of constructing a specific gravity bottle upon the design of a thermometer by having a long tube stopper was carried out for the writer many years ago by an expert Paris glass blower, but it had no provision for expansion of the liquids without loss, and was too tall for any ordinary balance. The design was improved and carried out better by the writer in 1884 (see *Ephemeris*, **2,** 529), and was still further improved in 1889 (see *Ephemeris*, **3,** 1162–1166). There was, however, still a recognized defect, in want of room for the expansion of liquids in warming up to room temperatures before weighing, and in so limiting loss by evaporation during expansion and weighing, that a practically constant weight could be attained. This last improvement seems to fairly satisfy these conditions, and in practice answers very well.

The bottle is an ordinary 500 cc. flask old enough to have reached its maximum contraction, and so large that errors of reading and weighing are well within the design of reaching the sixth decimal place with tolerable accuracy. A flask was selected with narrow neck, and the graduation mark low in the neck. The neck was cut off just above the mark and was thickened and shaped for stoppering. A piece of barometer tubing fifteen to twenty cm. in length was expanded into a long stopper at one end and carefully ground into the flask neck. Then 500 grams of recently boiled distilled water was put into the flask and stopper and the whole cooled in an iced bath to 4° C. (39.2° F.). The water in the tube stopper should then be near the lower end, but if not, the end is ground off until the meniscus for 4° C. is just above the ground part of the stopper. The 500 grams of water and the 4° C. temperature being then again accurately adjusted, a mark was made at the lower limb of the meniscus. The bath was then warmed up to 25° C. (77° F.) and when the water had ceased to rise in the tube after the temperature of the bath had been held constant for half an hour, another mark was made, the tube being of such a caliber that the two marks should be about ten cm. (four in.) apart. Any excess in the length of



APPARATUS FOR SPECIFIC GRAVITIES OF LIQUIDS.

the tube above the upper mark was then cut off, and a liberal section of a very much larger tube was put on. This larger portion was about 2.4 cm. (one in.) in diameter, and about seven cm. (2.75 in.) long, drawn into a narrow neck at the top, and the neck fitted with a flat ground glass stopper which had a capillary passage through its center. The tube portion of the stopper was then graduated in fifty equal subdivisions, the scale beginning with 0 at the 4° C. point, and extending to fifty at the 25° C. point. These subdivisions are easily and accurately read to half divisions so that the scale is read to hundredths. Then upon accurate readjustment it was found that the reading with recently boiled distilled water was one—that is $\frac{1}{100}$ of the scale from 4° C. to 25° C. The reading at 10° C. was six, or $\frac{6}{100}$ of the scale. At 15° C. the reading was twenty-five. At 15.6° C. (60° F.) it was twenty-eight. At 20° C. it was fifty-seven, and at 25° C. (77° F.) it was 101. These readings illustrate in a very interesting way the rapidly increasing ratio of expansion for water. Without correction for expansion of the flask the increase in volume for 25° C. (4° to 25°) is just 100 divisions of scale, giving a mean value of 4.76 + divisions to each 1° C. But the expansion for the first six degrees (4° to 10° C.) is only six divisions of scale, or one division to each 1° C. while the expansion for the last five degrees (20° to 25° C.) is forty-four divisions, or 8.8 divisions to each 1° C. This scale adapts the flask to taking specific gravities at all the ordinary standards of temperature, and adapts it to correction by readjustment at any time in case of contraction of the glass by age. It also enables the observer to watch the changes in height of the column of liquid and to know accurately when it ceases to rise or fall. When accurately adjusted the weights were taken on a fine balance to 0.0001 gram when chemically clean, and also when the stopper was lightly lubricated with soft paraffine,—these tares being etched upon the bottle, and all the readings of the standard water being entered in the laboratory note book.

BOTTLE IN USE.

The bottle, properly filled, and loaded with a leaden collar to steady it, was always used in a glass water-bath, the water of the bath being kept above the level of the reading lines, and

these read through the glass and water. The thermometer was suspended in the bath with the bulb opposite to the upper part of the bottle, and was also read through the glass and water; and a wooden stirrer was used to keep the water uniform throughout. When the temperature of the bath had been held steady to the required temperature for ten minutes after the column in the tube had ceased to rise or fall, the adjustment to the required graduation mark was made by passing the long capillary pipette and the very narrow strip of blotting card paper, shown in the cut, down through the tubulure in the stopper. In order to know how nearly the temperature of the liquid in the bottle agreed with that of the water of the bath, a pair of compared thermometers were used, and it was found that with either water or alcohol in the bottle, the temperatures were in accord to about 0.05° C. after all perceptible change in the column had ceased, and the bath had been held constant for five minutes; and upon this experience ten minutes was adopted as the uniform time of waiting before the final adjustment. Then it was found that no constant weight of bottle and contents could be obtained so long as these differed more than one or two degrees from the room temperature of scale-case and weights, and therefore a second bath was used after adjustment to bring them accurately to room temperature, and then they were allowed to stand on the balance with the case closed until they reached a fairly constant weight. As all this management required time, it became necessary to know whether there was any material loss in weight between the time of adjustment and the final weighing, and on trial it was found that with water the bottle and contents, either in the bath or out, did not alter perceptibly in weight within forty-eight hours, and with the alcohol under the same conditions, the loss in a warm room was not greater than 0.04 gram in forty-eight hours, or less than one milligram per hour. This loss was therefore neglected as being well within the sphere of unavoidable errors.

The weighing was done upon a large balance sensitive to 0.002 gram, and this was readjusted for each accepted critical weighing. The weights used were of brass, of a specific gravity of about 8.383, with platinum fractions of a gram. They were not stand-

ardized, but as they agreed among themselves, and as all the weighings were made with the one set, this gave no source of error. The critical weighings were made with the barometer not far from 760 mm. (thirty inches), and no correction for barometer was attempted. All weighings were made against water at the same temperature taken as unity, and therefore corrections for expansion of glass were avoided. Neither were any corrections attempted for the use of brass weights, because such corrections take the results out of the range of ordinary daily practice and tend to confusion rather than to practical utility. As the weighing was done to 0.002 gram in a 500-gram bottle the results are stated to the sixth decimal place, and although the sixth decimal cannot be trustworthy, even to twice the indicated value, it is still believed to be of more value than if expressed in the nearest unit of the fifth place. In view of the rapidly increasing importance of specific gravities of liquids, it is very desirable to give a more definite value to this sixth decimal place. The writer has 100-gram specific gravity bottles of the same construction which can be easily weighed on a fine balance to 0.0001 gram, but as yet the measuring or volume adjustment cannot be brought up to this high degree of precision.

LIME PERCOLATOR.

It was confidently expected that a more perfect dehydration might be obtained by often repeated percolation through lime than by the former process of prolonged shaking with lime, provided the percolation could be done without air contact, as was not done in the processes of the former paper.

A very large calcium chloride jar, holding almost a kilogram of lime, with large chamber below, was nearly filled with strata of granulated quick-lime alternately fine and coarse, the whole supported at the contraction below on a layer of glass wool. At the top a rubber stopper with two perforations was used, supplied with two glass tubes bent twice at right angles, the short leg projecting just through the stopper, while the long leg extended downward outside the jar. The tubulure of the chamber below was fitted with a rubber stopper of one perforation supplied with a glass tube, bent downward within so as to draw all the liquid

out, and bent upward without to receive rubber tubing. The object was to deliver the alcohol on top of the lime, draw it through the lime by means of the Sprengel water-pump, and then draw it out of the chamber below the lime to be again passed on top, and again through the lime, and this without contact with any air that had not been thoroughly dried. Near the lime percolator on each side was placed a bottle of two liters' capacity, each fitted with rubber stoppers supplied with a long and a short tube, the short tube passing just through the stopper, and the long one going to the bottom and so bent that its end reached the very lowest point in the bottle. A little farther off from the percolator on one hand was a large apparatus for drying the inspired air, and on the other hand the Sprengel water-pump. In the drying apparatus the air entered through a tube filled with caustic baryta, then bubbled through concentrated sulphuric acid and then ascended through a large jar of calcium chloride to the outlet. By means of rubber tubing and glass T and Y tubes these various parts were so connected that by means of screw pinch-cocks some passages could be stopped and others opened to the effect that the alcohol was easily and conveniently drawn from one bottle upon and through the lime into the other bottle, well dried air taking its place, until the bottle was empty and its fellow on the other side full. Then the closed pinch-cocks were opened, and the open ones closed and the alcohol was drawn back through the percolator to the other bottle, and so on. With a partial vacuum of only about 100 mm. (four inches) about four of these percolations were made in a day, and generally the alcohol was passed through about twenty-five times before being distilled.

APPARATUS FOR THE DISTILLATIONS.

The distillations were all made in one arrangement of apparatus. A round bottomed flask of about two liters' distilling capacity was held on a round bottomed water bath over a gas flame. The flask was fitted with a good cork that had been well dried by immersion in hot paraffine. This cork was perforated for a small tube through which to aspirate dry air, or the charges of alcohol, and also for the large end tube of a Hempel tube

about thirty-five cm. (13.7 inches) in total height, the passage of this tube through the cork being made air-tight by means of paraffine. The Hempel tube was then three-fourths filled with small glass marbles, and from the end of this tube, in the flask, a thermometer was hung. In the bottom of the flask, to prevent explosive boiling, was put a liberal supply of recently ignited fragments of clay, tobacco pipes, and platinum scraps. From the top of the Hempel tube a vapor tube of liberal size, bent twice at right angles, led to the condenser. The condensing tube was of thin glass, about seventy cm. (27.8 inches) long, turned up at one end to receive the vapor tube from the Hempel and bifurcated at the other end, each branch having a well ground glass stop-cock, well lubricated with soft paraffine—the ends of the branches beyond the stop-cocks being contracted and turned down. About fifty cm. (19.75 inches) of this tube passed water-tight through a tinned iron trough capable of holding a good supply of broken ice and water. The two terminal ends of the condenser were connected by rubber tubing with short straight pieces of glass tubing which passed through one hole of two two-hole rubber stoppers. The second hole of each stopper was fitted with a bent glass tube, and these were connected by rubber tubing with the aspiration tube of a competent Sprengel water-pump. The two rubber stoppers, through which the small glass tubes passed, fitted all the specific gravity bottles, and the arrangement admitted of distilling into one or the other bottle at will, and of changing bottles without the contact of much air, without interruption and without breaking the vacuum. The distillate was usually divided into seven fractions for each distillation. The first two in 100-gram specific gravity bottles, the third in the 500-gram specific gravity bottles, the fourth, sixth and seventh in 100-gram bottles, and the fifth in a 500-gram bottle.

In making the distillations the boiling was commenced under a partial vacuum of about 300 mm. (twelve inches), and without condensation in order that the pump might draw off all dissolved air and gases, and any liquids of low boiling point. Under this diminished pressure the boiling always began at about 50° C. (122° F.), and the temperature rose steadily to

about 67° C. (152.6° F.) before any considerable vapors went beyond the Hempel tube. Then the ice and water were put in the condenser trough, and the first 100-gram specific gravity bottle was filled under this partial vacuum. Then the vacuum was allowed to run down to about twenty-five mm. (one inch) and the remainder of the distillation was conducted under that slight minus pressure.

THE ORIGINAL ALCOHOL.

The following trials were all made with an alcohol that was always at hand in great abundance. It was believed to be quite free from all impurities excepting water and to contain only about 0.1 per cent. of water. The specific gravities taken with much care were as follows: At $\frac{4^\circ}{4^\circ}$ C., 0.802818; at $\frac{10^\circ}{10^\circ}$ C., 0.798018; at $\frac{15^\circ}{15^\circ}$ C., 0.794258; $\frac{15.6^\circ}{15.6^\circ}$ C., 0.793824; at $\frac{20^\circ}{20^\circ}$ C., 0.790714; at $\frac{25^\circ}{25^\circ}$ C., 0.787332. Provided the specific gravities given by Drinkwater, Fownes, Tralles and Mendelejeff were taken with as much accuracy as these now given here, this is the alcohol that, up to the date of these investigations, has been considered to be anhydrous, and it is the basis of all the best tables.

VALUE OF SPECIFIC GRAVITY IN PERCENTAGE.

Early in the investigation at this time, it became desirable to know with more accuracy than heretofore, the percentage value of specific gravities where there is so very little water present. To determine this point a portion of the original alcohol above described was used, and 1345.174 grams was carefully weighed off. To this portion 2.696 grams of distilled water was added giving a dilution of almost exactly 0.2 per cent. and the mixture was allowed to stand twenty-four hours, when the specific gravities were taken.

Specific gravity	At $\frac{4^\circ}{4^\circ}$ C.	At $\frac{10^\circ}{10^\circ}$ C.	At $\frac{15^\circ}{15^\circ}$ C.	At $\frac{15.6^\circ}{15.6^\circ}$ C.	At $\frac{20^\circ}{20^\circ}$ C.	At $\frac{25^\circ}{25^\circ}$ C.
Original alcohol	0.802818	0.798018	0.794258	0.793824	0.790714	0.787332
0.2 per cent. dilution	0.803434	0.798662	0.794932	0.794500	0.791362	0.787938
Difference for 0.2 per cent.	0.000616	0.000644	0.000674	0.000676	0.000648	0.000606
" " 0.1 " "	0.000308	0.000322	0.000337	0.000338	0.000324	0.000303

If these differences were interpolated and plotted they would give a form of curve for alcohol not hitherto known to this writer.

Next this portion of diluted alcohol was passed through the

lime percolator twenty-five times in six days to ascertain what proportion of this water might be abstracted by this treatment, and how near to a constant specific gravity or boiling point such alcohol would be. It had been recognized in the former investigations that a thermometer in the boiling alcohol was not an accurate indicator of the boiling point. In a clean glass flask the boiling was so nearly explosive that differences of a degree were often noticed within a very few seconds. Even with fragments of clay tobacco pipes and platinum scraps in the vessel the thermometer would rise and fall enough from time to time to preclude accurate observation of the constancy of the boiling point. The specific gravities of the fractions of distillate were, however, much better indications of the boiling point, since if these were constant, the boiling point would necessarily be also constant, and until these fractions were constant in specific gravity they could not be uniform in boiling point, and as long as they were not constant they could not consist of alcohol alone. That is, any mixture of two liquids as different in boiling points as alcohol and water could not distill at a constant boiling point, nor yield a distillate of constant specific gravity; and now it is known that a tenth of one per cent. of water in alcohol gives a very decided difference in specific gravity. The specific gravities given by the seven fractions of distillate from the portion of diluted alcohol that had been passed twenty-five times through the lime percolator were as follows, in the order in which they were received, all taken at $\frac{15.6^{\circ}}{15.6^{\circ}}$ C.: First, 0.79414; second, 0.79403; third, 0.79402; fourth, 0.79400; fifth, 0.79392; sixth, 0.79400; seventh, 0.79404. Then all the fractions were put together and the specific gravity of the mixture was 0.793954.

These results are accepted as proving conclusively that no part of the distillate was anhydrous alcohol, and that about 0.16 per cent. of the water added was taken out by the lime, leaving about 0.04 per cent. of the water put in that was not taken out by the lime.

DEHYDRATION BY METALLIC SODIUM.

Many times in the literature of late years metallic sodium has been recommended for the detection of water in alcohol, and for depriving alcohol of the last traces of water. But such recom-

mendations were not easily understood in view of the fact that at ordinary temperatures sodium decomposes alcohol rather rapidly. It might, however, also abstract water. Then, too, the well-known influence of cold in modifying chemical action might be available, so it was concluded to try the effects of sodium at low temperatures. About two liters of the alcohol was well cooled in a bath of snow and salt to about 17° C. (1.4° F.) and was held at that temperature or lower for about forty-eight hours. Throughout the day time of this period small pieces of sodium, two or three grams at a time, were dropped into the alcohol with the least practicable admission of air, and this was repeated five or six times a day, so that an aggregate of about forty grams of sodium was used. The sodium, instead of floating, went at once to the bottom, and the reaction was very moderate. This alcohol was then carefully distilled off from the sodium ethylate, the distillate being received in fractions, which varied considerably in specific gravity. The lowest fraction was, however, at $\frac{15.6°}{15.6°}$ C., 0.793713, and the average of the fractions was not below the 0.793824 of the original alcohol. This negative result is given because it may possibly save others from going over this ground.

Experiment 1.—A portion of good quick-lime was slaked with just enough water to make a fine dry powder. This was put into the center portion of a piece of three-inch wrought iron pipe, the two end portions being filled with quick-lime. Caps being screwed loosely on the ends the pipe was subjected to a dull red heat during four hours, and then cooled over night. About 500 grams of this lime was put into a bottle of 4.25 liters' capacity, and, 2.5 liters of the original alcohol having been poured upon it, the bottle was shaken half an hour at a time, in a mechanical shaker, during an aggregate of sixty hours in five weeks. The alcohol was then filtered off from all traces of lime, with as little air contact as practicable, and was aspirated into the distilling flask and distilled. The specific gravities of the seven fractions taken at $\frac{15.6°}{15.6°}$ C. were: First, 0.793851; second, 0.793839; third, 0.793680; fourth, 0.793701; fifth, 0.793679; sixth, 0.793400; seventh, 0.793701; an eighth fraction was 0.793620.

The sixth fraction of this series was rejected as being too low, and quite out of accord with the other elements of the series, but all the other weighings were trustworthy.

Experiment 2.—The mixed fractions from experiment 1 were passed through the lime percolator about ten times, and then again distilled, when the fractions gave the following specific gravities at $\frac{15.6°}{15.6°}$ C.: First, 0.793960; second, 0.793801; third, 0.793582; fourth, 0.793639; fifth, 0.793606; sixth, 0.793576; seventh, 0.793549; second weighing of the seventh fraction gave 0.793499. At the first weighing it had not reached a constant weight, while at the second weighing, thirty minutes later, there may have been a very slight loss.

Experiment 3.—A fresh portion of the original alcohol was passed twenty-five times through the lime percolator, and distilled with the following results: First, 0.793960; second, 0.793811; third, 0.793639; fourth, 0.793582; fifth, 0.793576; sixth, 0.793561; seventh, 0.793499. This last weighing, although reached here for the second time (see above), must be received with doubt, because it is not in accord with the other elements of its series.

When these results are carefully examined it will be seen that with better apparatus and better management, the results of the former investigations were not quite reached. If 0.000338 be the specific gravity value of 0.1 per cent. in alcoholic strength, then the former results, which reduced the specific gravity from about 0.793824 to 0.793500, or 0.000324, indicated a difference in strength of 0.09585 per cent.

Taking the mean of three trustworthy readings of this paper, namely 0.793549, 0.793576 and 0.793561, which mean is 0.793562, and subtracting it from the original alcohol, 0.793824, the difference is 0.000262 and this is equivalent to 0.07751 per cent.

The writer was unable to carry through the design of this paper for want of time and leaves one or two methods of dehydration that have occurred to him, still untried, and he also very much regrets having to leave, for the present, a thorough repetition of the former successful process of nine years ago. It seems to the writer very certain that up to the present time no really anhydrous alcohol has been obtained, and therefore with the

present better apparatus and management, and especially by prolonged contact with the lime, through the old process, better results might reasonably be expected.

PYROXYLIN, ITS MANUFACTURE AND APPLICATIONS.

By Walter D. Field.

Received June 17, 1893.

PART I.

BY the term pyroxylin is understood the soluble nitric ethers of cellulose, namely, the di, tri, tetra, and penta-nitrates. From the date of the use of pyroxylin in photography by Scott Archer in 1851, the number of its uses has increased until, at the present time, tons of the lower nitrates of cellulose are produced yearly. With this increase of production improved methods of manufacture have been evolved.

So general has become the use of this material that to-day it is to be found everywhere. In the form of celluloid it is before us constantly. As a varnish it is used on penholders, pencils, silver and brass ware. Articles are bronzed with it as a medium. An artificial leather has been produced with it, many thousands of yards of which have found a ready market. These applications are all made, with the exception of celluloid, by the use of a solution of pyroxylin.

The first part of this paper pertains to the fibers and their selection for the purposes of nitration; the second part to the processes used for nitrating. The third and fourth parts will treat of the various methods of washing and drying the pyroxylin and of its solvents and uses.

Selection of the Fiber.—Cotton fiber, wood fiber, and flax fiber in the form of raw cotton, scoured cotton, paper, and rags are most generally used and give the best results. The fibers differ greatly in their manner of nitrating, a difference due undoubtedly to their physical structure; hence a given fiber demands a method of nitrating suitable for that particular fiber.

The cotton fiber is a flattened hollow ribbon, or collapsed cylindrical tube, twisted a number of times, and closed at one

end to form a point. The central canal is large and runs nearly to the apex of the fiber. Its side walls are membraneous.

In regard to the influence of the structure of the cotton on nitration, F. Nettlefold (*Chemical News*, **55,** 306) says: "It will be readily understood that the thin side wall tubes of the cotton fibers are readily penetrated by the mixed acids, and, consequently, the highest nitration results."

Wolfram (*Ding. Poly. Jour.*, **230,** 45) says: "Concentrated acids give with cellulose from various sources the same final product, but dilute acids nitrate under the same conditions—cotton the most readily, then hemp, paper, straw, and linen."

Hardwich (*Manuel Phot. Chem.*, 136) recognizes the varying effects produced by the same strength of mixed acid on different fibers. He ascribes this difference to a difference in composition of the fiber and not to a difference in structure. In this conclusion he was undoubtedly wrong.

In the flax fiber the walls are comparatively thick, the central canal small; hence it is to be presumed that the nitration must proceed more slowly than in the case of cotton, which has large central canal and thin walls. The New Zealand flax gives the most perfectly soluble nitrates of any of the flaxes.

Hardwich (*loc. cit.*) says: "When a rather concentrated nitro-sulphuric acid is used cotton gives a glutinous collodion and calico a fluid collodion. In another acid weaker than the first the cotton succeeds well while the calico dissolves instantly. The difference in the action appears to depend principally on the thickness of the fiber. Calico produces pyroxylin of the fluid kind and is partially dissolved because the nitric acid in acting on the outside portion of the closely twisted fiber is reduced in strength, and hence the interior of the fiber is left more nearly in the condition of zylodine." By closely twisted fiber Hardwich refers to the thread from which the fabric is woven, and with this understanding his explanation of the action of the acids is clear.

One of the largest manufacturers of pyroxylin for the industrial arts uses the "Memphis Star" brand of cotton. This cotton is an upland cotton and its fibers are very soft, moist, and elastic. Its color is a light creamy white. This color is re-

tained by the cotton after nitration. The staple is short, and the twist inferior to other grades, the straight, ribbon-like filaments being quite numerous. They use this cotton carded, but not scoured. This brand of cotton contains a large quantity of half and three-quarter ripe fiber, which is extremely thin and transparent, distributed throughout the bulk of the cotton (Monie, *Cotton Fiber*, 67). This is a significant fact when it is known that from this cotton an extremely soluble pyroxylin can be produced.

Pyroxylin of an inferior grade, as regards color only, can be produced from the cotton wastes of the trade. These wastes are scoured, which renders them clean and suitable for nitrating. Paper made from the pulps of the sulphite and sulphate processes is capable of yielding a very soluble pyroxylin. It can be nitrated at high temperatures and still yield an increase in weight. The manufacturers of celluloid in this country use a tissue paper made of the flax fiber, and two to three one-thousandths of an inch in thickness. The paper is cut into squares of about one inch before nitrating.

Mowbray (United States patent No. 443,105, Dec. 3, 1890) says that a pure cotton tissue paper less than one five-hundredth of an inch in thickness, thin as it is, takes on a glutinous or colloid surface and thus requires some thirty minutes or thereabouts to enable the immersion to take place. With a thicker paper only the surface of the paper would be nitrated and the body of the paper would be unacted upon.

Mowbray (U. S. patent No. 443,105) recognizing the importance of the structural form of the cellulose on the nitrating process, uses a fiber which has been saturated with a solution of nitrate of soda, then dried slowly, claiming that the salt crystallized in the fiber or enters by the action termed osmose and opens up the fiber to the action of the acid bath. He also says he may use "any salt crystallized, or water crystallized by freezing in the cells of fibrose cellulose." Such a preparation of the fiber is not at all necessary when using a high temperature for nitrating. It might yield with some cottons a very soluble pyroxylin, when low nitrating temperatures are used. H. De. Chardonnet (English patent No. 19,560, 1891) heats the cellu-

lose intended for nitration to about 150° C. and claims to increase the solubility of the subsequently formed pyroxylin. The heating of the cellulose could have had no other effect than that of drying the fiber before nitration, and hence a pyroxylin more perfectly soluble in the ethereal solvents he was using in his testing.

Henly and Spill (English patent No. 1017, 1870) use "esparto grass in a finely divided condition (which is the novel and indispensable condition of the operation)."

Dietz and Wayne (U. S., patent No. 133,969, 1872) use ramie, rheea, or China grass, for producing a soluble pyroxylin and say in regard to it that "Pyroxylin made from ramie will always be of uniform strength and solubility. In making collodion from this pyroxylin a much smaller quantity of solvent is required than for pyroxylin made from cotton." It is to be doubted that ramie will yield a more soluble pyroxylin than cotton. The author's experience is entirely to the contrary.

Such is the influence of the physical form of the fiber on the process of nitration that when flax fiber and cotton fiber are nitrated with acid mixtures of exactly the same strength, and at the same temperature the solution of the first is glutinous or thick and the second fluid or thin. By simply nitrating at a higher temperature than the cotton, the flax will yield a pyroxylin giving an equally fluid collodion.

The presence of chlorine in the fiber must be carefully avoided

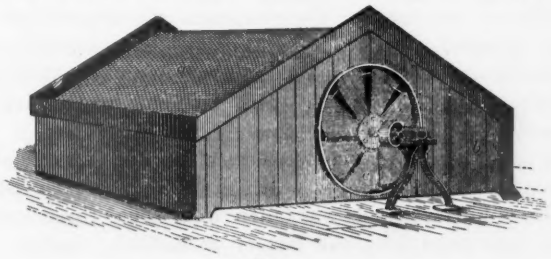


FIG. 1.

as such a fiber will yield a pyroxylin which is acid and which cannot be washed neutral.

The removal of all moisture from the fiber previous to nitra-

tion is essential to the production of a carefully prepared pyroxylin. This drying is best carried out on the form of drier used in drying wool. The plan of such a drier is given in Fig. I. The surface *a* is covered with wire cloth, having a half inch mesh, and should be galvanized.

Heater coils can be placed within the frame *b* and the hot air forced through the fiber by means of the rotary fan *c*.

If the fiber is found to need scouring to make it fit for the process of nitration, it is best scoured by those who make a business of this branch. Absorbent cotton is produced on such a large scale at the present time, that the manufacturers will sell it at a very low figure, in order to keep their plants in operation. The author obtained the fiber used by him for several years from this source.

[CONTRIBUTIONS FROM THE CHEMICAL DIVISION, U. S. DEPARTMENT OF AGRICULTURE, NO. 6; SENT BY H. W. WILEY].

THE PREDOMINANT ORGANIC ACID IN SORGHUM JUICE.[1]

BY OMA CARR.

Received June 1, 1893.

IN the work of the Department of Agriculture during the past few years attention has often been called to peculiarly tenacious and difficultly soluble incrustations forming upon the tubes of the evaporating apparatus of the sorghum-sugar houses of Kansas. Owing to the formation of these incrustations and the difficulty of removal the processes of manufacture are greatly impeded. Dr. Wiley collected a large amount of this scale at Medicine Lodge and directed me to make a study of its composition and properties. The following paper gives the results of a brief study of the scale.

The scale examined was in pieces of varying sizes, retaining the contour of the tubes from which it had been removed. A quantity of fifteen pounds was taken from some three hundred pounds forwarded from the factory at Medicine Lodge. The scale as prepared for examination consisted of a powder sufficiently fine to pass an eighty mesh sieve.

The analyses of two samples of the scale, one designated

1 Read before the Chemical Society of Washington, May 11, 1893.

"Special," selected by Dr. Wiley for its size and cleanliness, and "Ordinary," representing the whole quantity removed, gave the following results:

	"Special."	"Ordinary."
Moisture	13.31 per cent.	14.21 per cent.
Magnesium oxide	15.93 " "	17.70 " "
Calcium oxide	7.27 " "	9.80 " "
Carbon dioxide	0.29 " "	2.76 " "
Fe_2O_3, Al_2O_3, P_2O_5	6.22 " "	4.10 " "
Silica	2.72 " "	1.75 " "
Organic	54.40 " "	49.31 " "
Undetermined		0.37 " "
	100.14 " "	100.00 " "
Carbon	23.67 " "	22.29 " "
" per cent. of organic	43.51 " "	45.20 " "

These data call for no special comments, aside from noting that the common scale has lain long enough for the absorption of considerable carbon dioxide, and that the elimination of this constituent would secure a greater uniformity between the two samples.

Solubility.—The ground material, one gram in quantity, was boiled an hour and a half with fifty cc. of the solvent, filtered hot upon a weighed asbestos film in a Gooch crucible, washed with alcohol and ether, dried at 105° and weighed. For this determination the "Special" sample was used, and it may be assumed that the slightly different composition of the two samples would cause but slight variation in the results.

Solvent.	Per cent. soluble.	Per cent. Inorganic insoluble.	Per cent. Organic insoluble.
2 per cent. HCl	95.67	2.85	1.48
5 " " "	95.00	2.78	1.32
10 " " "	95.97	2.60	1.43
20 " " "	95.87	2.59	1.54
Mean	95.85	2.70	1.45
2 per cent. H_2SO_4	94.48	3.67	1.85
5 " " "	95.81	2.56	1.63
10 " " "	95.85	2.74	1.41
*20 " " "	81.03	16.29	2.68
Mean	91.79	6.32	1.89
** " (2nd)	95.38	2.99	1.63
2 per cent. NaOH	67.15	21.58	11.27
5 " " "	62.45	24.68	12.87
10 " " "	59.85	29.47	10.68
20 " " "	60.35	26.29	10.20
Mean	62.45	26.29	11.26

*Boiled only one hour. **Omitting the twenty per cent. determination.

It is seen from these results that hydrochloric acid has the highest solvent power of the three tried. Subtracting the organic insoluble from the total insoluble, the difference corresponds almost exactly to the per cent. of silica, showing that, aside from the small percentage of organic matter, the hydrochloric acid dissolves every thing but the silica. It is notable that the solvent power of hydrochloric acid depends very little upon its concentration.

The sodium hydroxide dissolves less than either acid, its power decreasing with increase of concentration. The portion insoluble in sodium hydroxide contains no silica.

In cleaning incrusted tubes, the above results, as well as good practice, suggest a procedure in which the use of hydrochloric acid precedes that of the lye.

As the work on the scale was done principally with the view of isolating and distinguishing the chief organic acids present, the following plan was pursued:

Finely divided scale (passing eighty mesh) was added to boiling sulphuric acid in ten per cent. solution until solvent action ceased and filtered hot, the filtrate being perfectly clear and transparent. The filtrate was cooled and two volumes eighty per cent. alcohol added. The calcium and magnesium sulphates precipitated were removed by filtration. The alcohol was expelled from the filtrate and the latter considerably reduced in volume by evaporation. There was added now a very strong alcohol (five volumes) and the white precipitate allowed to settle. It was decanted and washed with alcohol. The precipitate was purified by dissolving in water and repeated reprecipitation by alcohol. Finally from the solution in water free of carbon dioxide it was precipitated with a carbon dioxide-free solution of acetate of lead. This product was purified by repeated decomposition with hydrogen sulphide and reprecipitated with lead acetate. The final precipitation yielded my salt (I), or "scale salt," the combustion of which gave:

	Per cent.
Carbon	13.884
Hydrogen	1.210
Lead	58.420
Oxygen (difference)	26.486
	100.000

The salt contained 2.46 per cent. moisture, eliminating which would give the following numbers:

	Per cent.
Carbon	14.23
Hydrogen	0.94
Lead	62.11
Oxygen	22.72
	100.00

These figures correspond to the formula:

$(C_6H_5O_7)_2Pb_3$, or tri-plumbic citrate.

After digestion with sulphuric acid the insoluble residue, undried and unwashed, subjected to analysis, gave the following numbers:

	Per cent.
Moisture	6.83
Calcium oxide	13.92
Fe_2O_3 and Al_2O_3	1.70
Silica	4.26
SO_3	62.43
Organic	10.47
Undetermined	0.39
	100.00

The SO_3 present was combined, twenty per cent. with calcium (42.77 per cent. sulphate), and 1.98 per cent. with the Fe and Al (3.88 per cent. sulphates). The rest was free H_2SO_4. Eliminating the SO_3 we have:

	Per cent.
Moisture	18.18
Calcium oxide	37.05
Fe_2O_3 and Al_2O_3	4.52
Silica	11.34
Organic	27.87
Undetermined	1.04
	100.00

Reducing to a water-free basis these data become:

	Per cent.
Calcium oxide	45.28
Fe_2O_3 and Al_2O_3	5.52
Silica	13.86
Organic	34.06
Undetermined	1.28
	100.00

Reducing original scale to water-free substance and comparing its analysis with that of the residue after digestion we have:

	Original.	Residue.
Magnesium oxide	20.63 per cent.	....
Calcium oxide	11.41 "	45.28 per cent.
Carbon dioxide	3.22 "	....
Fe_2O_3 and Al_2O_3	4.78 "	5.52 "
Silica	2.04 "	13.86 "
Organic	57.48 "	34.06 "
Undetermined	0.43 "	1.28 "
	100.00 "	100.00 "

The silica having remained unaffected by the digestion, we may place the residue on the same plane as the original material by decreasing the percentages in the residue in the ratio of the respective percentages of silica. Analyses so corrected are as follows, calculated to water-free substance:

	Original.	Residue.
Magnesium oxide	20.63 per cent.	....
Calcium oxide	11.41 "	6.66 per cent.
Carbon dioxide	3.22 "	....
Fe_2O_3 and Al_2O_3	4.78 "	0.81 "
Silica	2.04 "	2.04 "
Organic	57.48 "	5.01 "
Undetermined	0.43 "	0.13 "
Removed	....	85.35 "
	100.00 "	100.00 "

The organic matter has fallen from 57.48 per cent. to 5.01 per cent., 52.47 per cent. being removed, together with 20.63 per cent. MgO. Dividing the per cent. of the removed organic matter by the molecular weight of both aconitic and citric acid, we have:

$$52.47 \div 210 = 0.205 = \text{citric}$$
$$52.47 \div 174 = 0.301 = \text{aconitic}$$
$$20.63 \div 120 = 0.172 = MgO$$
$$20.63 \div 100 = 0.206 = 2\tfrac{1}{2}\ MgO$$

As, however, the organic matter of the water-free original material contained 45.19 per cent. carbon, and as citric acid contains but 37.7 per cent., other organic matters are present containing the difference between 45.19 per cent. and 37.7 per cent., or 7.49 per cent. Correcting the decimal given for citric

acid (0.205) by the equation 45.19 : 37.7 :: 0.205 : x, we find x to equal 0.171, or practically identical with the decimal for 3MgO.

Aconitic acid contains 41.84 per cent. carbon, and correction of the decimal by substitution of 41.48 for 37.7 in above equation brings the decimal to 0.278.

ACETIC ACID DIGESTION.

As with sulphuric acid, in this digestion the scale was added to boiling acid, the solution being arrested before discoloration commenced. The lead salt was prepared in the manner described for sulphuric acid, due precautions being taken to prevent formation of lead carbonate. Combustion of this salt resulted as follows:

	Salt II (acetic acid digestion).
Carbon	13.03 per cent.
Hydrogen	1.74 "
Lead	57.69 "
Oxygen (diff.)	27.54 "
	100.00 "

The salt contained 7.50 per cent. moisture, eliminating which we have:

	Salt II.	Theory tri-plumbic citrate.
Carbon	14.09 per cent.	14.42 per cent.
Hydrogen	0.89 "	1.00 "
Lead	62.37 "	62.15 "
Oxygen	22.65 "	22.43 "
	100.00 "	100.00 "

It would appear that this salt has slightly oxidized in drying. After digestion the insoluble residue contained:

		Moisture free.
Moisture	11.84 per cent.	....
Magnesium oxide	16.63 "	18.98 per cent.
Calcium oxide	9.74 "	11.05 "
Fe_2O_3 and Al_2O_3	2.43 "	2.72 "
Silica	2.39 "	2.71 "
Organic	54.26 "	61.54 "
Undetermined	2.72 "	3.00 "
	100.00 "	100.00 "
Carbon	19.49 "	22.11 "
Carbon per cent. of organic matter	....	35.93 "

From an equal weight of material much less of the lead salt was secured from the acetic acid digestion than from the sulphuric acid preparation.

For the purpose of comparison a pure tri-plumbic citrate was prepared, the combustion resulting as follows:

	Salt III (tri-plumbic citrate).
Carbon	13.96 per cent.
Hydrogen	1.33 "
Lead	59.76 "
Oxygen (diff.)	24.95 "
	100.00 "

The salt contained 3.59 per cent. moisture, elimination of which produces the following numbers:

		Theory tri-plumbic citrate.
Carbon	14.42 per cent.	14.42 per cent.
Hydrogen	0.93 "	1.00 "
Lead	61.98 "	62.15 "
Oxygen	22.61 "	22.43 "
	100.00 "	100.00 "

The three lead salts, corrected for uncombined water, were grouped thus in the following table and compared with the theoretical salt:

(I) From sulphuric acid digestion.
(II) From acetic acid digestion.
(III) Tri-metallic citrate of lead.
(IV) Tri-metallic citrate of lead, theory.

	I. per cent.	II. per cent.	III. per cent.	IV. per cent.
Carbon	14.23	14.09	14.48	14.42
Hydrogen	0.94	0.89	0.93	1.00
Lead	62.11	62.37	61.98	62.15
Oxygen	22.72	22.65	22.61	22.43
	100.00	100.00	100.00	100.00

From the above it is apparent that the lead salts are unquestionably citrates.

For further proof the acid was freed of lead by hydrogen sulphide, excess of the precipitant removed, and in aqueous solution the following reactions were obtained:

(I) With bichromate of potash, *coffee brown color.*

(II) Boiled with strongly alkaline potassium permanganate gave *green color.*

(III) Heated at 100° C. for an hour with ten parts sulphuric acid, *lemon yellow color.*

(IV) Characteristic reaction of *precipitation when heated in aqueous solution.*

Parsons (*Am. Chem. J.*, **4**, 39) gives the results of an examination of "scale from sorghum-sugar pans," and concludes that the "scale is a two-thirds metallic calcic aconitate (as this is a quotation we do not feel at liberty to change from Parsons' own words to "dicalcium aconitate") containing one molecule of combined water." Comparing Parsons' data with those obtained in these more recent researches it seems probable that the aconitic acid is a product formed in manufacture. The scale examined by Parsons was derived from pans in which the boiling was conducted under atmospheric pressure. The scale recently examined came from evaporators in which the boiling was done in vacuo. Inasmuch as citric acid when strongly heated loses one molecule of water and becomes aconitic according to the equation, $C_6H_8O_7 - H_2O = C_6H_6O_6$, it is probable that Parsons' scale formed under high temperature and prolonged heating may have suffered dehydration and become an impure calcium aconitate. It would appear also that the scale formed in the evaporating apparatus of sorghum-sugar houses contains little, if any, aconitate. At the same time it may be conjectured that the reported presence of aconitic acid in the refuse or final products of sugar cane houses, molasses, skimmings, etc., may be attributed to formation in the manufacturing processes and not to presence in the canes or juices.

A CRYSTALLINE CHROMIUM TUNGSTATE.

By Edgar F. Smith and Herman L. Dieck.

Received June 27, 1893.

POTASSIUM bichromate was brought together with an equivalent amount of pure tungstic oxide. Heat was applied to the mixture; fusion followed and the mass was held in this condition until the evolution of gas bubbles ceased. The temperature was never higher than necessary to maintain a liquid condition of the mixture. After cooling, the fused mass

was treated with water until the latter ceased to dissolve anything. Sodium hydroxide and ammonium hydroxide were next employed for washing purposes, and these finally displaced by pure distilled water. The well-washed substance was carefully dried at 100°. It showed a dark brown color, and when examined under the microscope revealed the presence of acicular, brown-colored crystals of orthorhombic outline; associated with these were green-colored crystals, which were apparently of the same crystallographic system. This mixture was exposed to the action of aqua regia for an hour; it was then filtered, washed, and dried. A re-examination of the brown residue showed that the green crystals had been removed and that the brown-colored body alone remained. It had been unaffected by the acid. Two weighed portions of the mixed crystals were treated with aqua regia, the acid solutions evaporated, the residues dissolved in water, and the aqueous solutions treated with ammonium hydroxide. Chromium hydroxide was precipitated. In the one case the ignited oxide equaled 2.89 per cent. and in the other 2.92 per cent. of the original mixture. A sufficient quantity of pure brown crystals was prepared for analysis. We first endeavored to effect their decomposition by fusion with sodium carbonate and sulphur. Several attempts demonstrated that this course offered little chance of a satisfactory analysis. Digestion with hydrofluoric acid gave a poor result; the acid failed to completely decompose the material. Portions of the substance were next ignited in a current of hydrogen, the residue then oxidized with aqua regia, evaporated to dryness and treated with ammonium hydroxide. Considerable tungsten was removed in this manner, but much remained with the chromium, from which it seemed almost impossible to free it. The mixture of tungstic and chromium oxides was ignited a second time in hydrogen and again treated with aqua regia. This operation was repeated until no more tungsten could be extracted. Combining the separate portions of tungstic oxide, their total quantity gave 88.37 per cent. WO_3. The chromium oxide, previously associated with this amount of tungstic oxide, equaled after careful purification, 11.75 per cent. This method of analysis having proved tedious and time-consuming, we de-

termined to try fluxing the brown-colored material with a mixture of pure sodium carbonate and potassium nitrate, as we had previously satisfied ourselves that it did not contain the alkali metals. The fusion was taken up with water, acidulated with hydrochloric acid, and evaporated to dryness. Tungstic acid separated; the residue was moistened with acid and again evaporated. It was then digested with acid and left pure tungsten trioxide which was filtered, washed, and weighed. It equaled 88.08 per cent. The chromium oxide in the filtrate, after precipitation and purification, equaled 12.10 per cent. Upon repeating this course with new portions of the substance we obtained results that agreed with those just given, so that we feel satisfied that they represent the true composition of the brown crystals. Taking the mean of the percentages of the two analyses given above as a basis of calculation we deduce as the most probable ratio, $1Cr_2O_3, 5WO_3$ or $Cr_2O_3.5WO_3$. The theoretical requirements of this formula are:

	Per cent.	
WO_3	88.40	
Cr_2O_3	11.59	
	Per cent. found.	
WO_3	88.37	88.08
Cr_2O_3	11.75	12.10

Tungstates of chromium that exist[1] are hydrous and have been prepared by double decomposition in the wet way. The salt we describe is, so far as we can ascertain, the first of its class.

UNIVERSITY OF PENNSYLVANIA,
June 15, 1893.

THE METHODS OF TESTING FATS AND OILS.

BY DR. ERNEST MILLIAU, DIRECTOR OF THE GOVERNMENT TESTING LABORATORY, MARSEILLES, FRANCE.

Read before the New York Section May 29, 1893.

THE difficulties of chemical analyses of fatty matters are greatly increased, not only by the slight differences which characterize the various glycerides, but also by the numerous cases of isomerism, and the phenomena of oxidation and fermentation which modify their molecular structures, the resins and the essen-

[1] *Fremy's Encyclopedie Chimique*, 3, 166.

tial oils which they contain, the foreign matters which spoil them, the composition of which varies infinitely, according to the nature of the soil from which the plant draws nourishment, the method of extraction, age, and a thousand other causes, needless to mention. Since the methods of communication have been facilitated and exchanges have become more numerous, the different markets of the world have been flooded with fatty matters presenting different characters, though of the same nature as those which industry and commerce have been accustomed to.

We have been led to seek methods of analysis for oils not by their impurities, which vary according to their origin, but by the examination of their principal constituents, which remain practically the same for the same species.

For example, it is now well known that the olive oil of Tunis, Morocco, and other provinces presents often the character of adulterated oils if analyzed without proper precautions or by old processes.

Accordingly most governments and large companies have modified their specifications. It is indispensable for obtaining precise results to submit the material to a purification, preceded and followed by a number of filtrations. By thus removing the impurities, which when present alter the results, we bring back the fatty bodies of the same species to a type which is always practically the same. To reach this end we work sometimes with washing by hot distilled water, sometimes by strong or dilute alcohol, sometimes we refine the fatty matter by the use of caustic soda lye, containing ten per cent. sodium hydrate, employed in the proportion of ten per cent. of the fat. The emulsion is poured upon a saturated solution of sodium chloride, and by the action of gentle heat, the different parts separate and the clear oil holding in suspension insoluble particles of soap rises quickly to the surface.

It is well to note that fatty matter, even when neutral, as has been demonstrated, dissolves notable quantities of alcohol, which must be eliminated if this method of washing has been employed. The nature of the operation can not be fixed definitely; it varies according to the nature of the impurities and the object

of the research. In determining the amount of volatile acids we must be contented with simple filtration and when examining the oils of the cruciferae we must not use caustic soda, which changes organic sulphur to soluble sodium sulphide. Having finished the preliminary operations we can, according to circumstances, operate directly on the neutral fat or the fatty acids prepared from it.

The fatty acids collected in the nascent state, that is to say when they rise to the surface in a pasty mass, have much stronger chemical affinities than when melted and dehydrated. This difference is particularly remarkable in the examination of cotton-seed and sesame oils, the black and red colorations obtained very clearly with a mixture of five per cent. upon the nascent fatty acids are not visible with the same fatty acids when melted.

That purification skillfully made does not destroy the chemical characters of the oil, is easy to verify by treating comparatively the same oil containing five per cent. of the fatty matter the presence of which we wish to demonstrate. The reactions of the added material are in general intensified by these different treatments.

By working upon the fatty acids of the neutral oils I have been able to demonstrate the purity of certain fatty matters which appeared adulterated and in which the presence of cotton-seed and sesame oils was indicated by the old processes, which I have been able to revise. (*Circulaire du ministre de la marine français, en date du 26 julliet, 1892. Rapport du Resident général de Tunisie, en date du 9 mai, 1892. Circulaires des ministres du commerce et de l'agriculture, etc.*)

I well know that in practice these preliminary operations may appear very tedious, but I do not advise their use except in cases where adulterations have been indicated by the usual methods, and it is desired to make the proof positive.

Besides I cannot understand why a chemist has always been expected in the analysis of such delicate materials as fats to discover an infallible reagent, a single drop of which will immediately turn olive oil green, arachide black, sesame red, etc., so that any inexperienced man could discover adulteration and

bring the perpetrator to justice. This requirement of the most difficult part of chemical verification, is absolutely incomprehensible and is only to be excused by the ignorance of those who ask it.

We will now proceed to the study of each oil in particular. We will pass rapidly over those which possess only a secondary interest so as to lay greater stress upon those which are the object of a great number of sophistications as for example, olive oil. But in the first place we will indicate rapidly the general processes physical and chemical which are used for the identification of the various fats.

GENERAL METHODS.

(1) *Specific Gravity.*—Is taken with accuracy by the Mohr balance, which is too well known to need description. It must be borne in mind that the specific gravity of the same fat varies greatly with individual samples, and this physical character is not sufficient to allow us to decide on purity or adulteration.

(2) *Action of Nitrous Fumes.*—We owe to a Marseilles gentleman, M. Poutet, the first serious process to recognize certain adulterations of olive oil. It was supposed especially to discover the presence of poppy-seed oil which at that time was a very frequent adulteration and for which the process gave good results.

It is based upon the transformation of the oleine to its isomeric modification elaidine by the action of nitrous fumes. It has been modified by other chemists, Messrs. Boudet, Fauré, and Cailletet. The process of M. Cailletet was most sure and simple and we use it in the following manner:

We take a tube ten cm. long, 2.5 cm. wide, in which are twenty cc. of the fat to be analyzed. Six drops of pure sulphuric acid at 66° B., are added, the tube shaken one minute, and then nine drops of nitric acid C. P. 40° B. are added, after which the tube is again shaken and then plunged in boiling water, where it is left exactly five minutes, after which it is cooled in a water bath at 8°–10° C., whence it is taken at the end of two hours, and the condition of the mass observed. It is well to note the different colorations obtained, first after the addition of the

sulphuric acid, second after the addition of nitric acid, third after removal from the water bath and fourth after chilling.

(3) *Sulphuric Saponification.*—M. Maumené proposed to observe the rise of temperature produced by rapidly mixing sulphuric acid with oil. The manner of applying the process in our laboratory is as follows: Fifty grams of the fat for analysis are weighed into a conical glass of 100 cc. capacity. The temperature is noted and ten cc. sulphuric acid 66° B. at the same temperature as the oil is added. The two liquids are stirred together for one minute. An accurate thermometer is then immersed in the upper portion of the mass, stirred slowly, and the maximum temperature noted. The initial temperature must be at least 20° C. In order to obtain exact results it is well to make several determinations and take their mean when the variation does not exceed 2° C.

To obtain the relative sulphuric saponification, we note the rise of temperature obtained with fifty grams of distilled water at the same temperature as the oil and ten cc. of the same sulphuric acid. The number of degrees obtained by the oil is multiplied by 100 and the product divided by the number of degrees obtained with water. This process has the advantage of giving results, nearly constant, with acids of somewhat different strength. I have tried the application of the sulphuric saponification with semi-solid vegetable oils by working at 2°–3° above their melting points, and the results have been very satisfactory, especially for palm-nut and cocoanut oil, because the differences between them and the fluid oils often amount to 45° or more,—as between palm-nut oil and sesame oil, for example.

(4) *Iodine Number.*—In the fatty bodies there are members of unsaturated series, as for example oleic acid, which can absorb into its molecule as many atoms of the halogens as there are lacking atoms of hydrogen for complete saturation.

The iodine number of olive oil varies between eighty and eighty-four, that of arachide or peanut oil is ninety-seven, while that of cotton-seed oil is 108. These differences then allow, within certain limits, the determination of the purity of the various fats. We apply the Hubl method as follows:

Solutions required:

	Grams per liter.
Alcoholic solution of iodine	50
Solution of sodium thiosulphate	24.8
" " mercuric chloride	60
" " potassium iodide	100

Five (5) grams of fatty acids are weighed out, diluted to 100 cc. with ninety-two per cent. alcohol, ten cc. of the solution are taken, and to it added twenty cc. of the iodine solution and fifteen cc. to twenty cc. of the solution of mercuric chloride. The flask is closed and allowed to stand three hours. Twenty cc. of the potassium iodide solution is added and the excess of iodine titrated. When the brown coloration begins to disappear a few drops of starch solution are added and then thiosulphate until the color disappears.

We will pass in silence the other indices, such as that of acetyl, which is of use only to discover castor oil.

(5) *Freezing Point.*—The freezing point is easy to determine by means of a thermometer and a freezing mixture.

(6) *Melting Points of Fatty Acids.*—The dry and melted fatty acids are sucked into a capillary tube. After solidification the tube is placed beside the bulb of a sensitive thermometer and immersed in a beaker of water, the temperature of which is raised very slowly. The reading of the thermometer is taken the instant the body passes from the solid to the liquid state.

(7) *Solidification Point of Fatty Acids (Titer).*—The dried and melted fatty acids are placed in a tube fifteen cm. long and two cm. in diameter, which is suspended in a wide-mouthed bottle with a perforated stopper. A thermometer graduated to tenths is inserted so that the bulb reaches the center of the material. At the moment solidification commences a circular movement is given to the thermometer, stirring the whole mass; the thermometer is then left at rest and carefully watched till the mercury ceases to rise, the reading then taken gives the point of solidification or titer conventionally adopted.

(8) *Saturation.*—We operate on five grams of the melted and dried fatty acid with a solution of normal caustic soda. The number of cc. absorbed gives the saturation number.

(9) *Solubility in Absolute Alcohol.*—We determine this in the

following manner: The fat is neutralized by agitating in a closed separating funnel for thirty minutes with twice its weight of ninety-five per cent. alcohol; after settling it is drawn off and the alcohol held in solution driven off at low temperature, after which it is agitated at 15° C. or at a few degrees above the melting point, if solid at 15° with twice its weight of absolute alcohol. A known quantity of this alcohol is evaporated, the residue weighed, and the quantity of oil dissolved by 1,000 grams of alcohol is calculated.

FLUID VEGETABLE OILS.

Olive Oil.—Olive oil, like other vegetable oils, is a mixture of neutral glycerides, oleine, stearine, palmitine, etc., and a variable quantity of free fatty acids. It is the edible oil *par excellence*, and the south of France owes a portion of its prosperity to the cultivation of the olive tree. This culture is to-day almost precarious in certain countries, though the consumption continually increases, and olive oil always enjoys the just title of uncontested superiority. We can only attribute these remarkable results to the adulterations made for the purpose of selling under the name of olive oil, seed oils, or adulterated olive oil. We would be easily freed from these adulterations if chemists had at their disposition absolutely sure means to recognize them. Unfortunately the similarity of composition and reaction of the various vegetable oils render the demonstration of their presence extremely difficult. In face of the prejudice caused by adulteration the greatest efforts have been made to solve the problem, and although the results have not been sterile, they have not yet reached a satisfactory solution. It is necessary to say that if certain adulterators succeed in selling seed oils disguised as olive, they reverse the problem for the chemist by making him seek olive oil in seed oil. I may remark that the fraud is not always so great; very often the proportion of seed oil is not higher than ten per cent. Consequently for our method of analysis to be considered absolutely good it must give results, without recourse to comparisons, which satisfactorily show five per cent. in a mixture. Under these conditions we are certain to discover adulteration when made in the proportion of ten per cent.

Let us rapidly review the general processes for the recognition of peanut, sesame, cotton-seed, and poppy oils in olive oil, and we must acknowledge that none of them demonstrate less than ten per cent.

Specific Gravity.—The specific gravity of olive oil varies from 0.915 to 0.918. We have 0.917 to 0.918 for undecorticated peanut, 0.921 for decorticated peanut, 0.923 for sesame, 0.921 to 0.924 for cotton-seed oil, 0.924 for poppy oil. These variations of density in the olive oil do not allow us to recognize a mixture with any degree of certainty.

Action of Nitrous Fumes.—When taken from the water bath, if the oil contains a fairly large proportion of peanut oil, it will appear wine red. Pure olive oil is, on the contrary, lemon yellow. When taken from the cold bath a complete solidification is observed with pure olive oil, which has the appearance of very light fresh butter. With a mixture of fifteen per cent. of all other oils there is no solidification. This method gives good means of detecting peanut and poppy oils in edible, but is of less use when applied to oils used for industrial purposes, which may not solidify though pure.

Sulphuric Saponification.—The rise of temperature of olive oil is 35°, the relative 94°. A rise of temperature above 35° usually indicates adulteration. Equal or lower results do not absolutely indicate purity because certain pure olive oils give only 31°, 32°, and 33°, and in consequence after the addition of seed oils show only 34°–35°.

Indices.—The iodine number varies from eighty to eighty-five, and in a certain measure helps us to discover seed oils. It likewise offers us a quantitative method with a mixture of known oils. The variation in composition of the same oil spoils its sensitiveness for determining mixtures from five to ten per cent.

We will not mention the other general processes, but will dwell particularly on the special reactions which characterize the presence of the three oils most frequently used to adulterate olive oil, to wit, peanut, sesame and cotton-seed.

These processes have a much greater analytical value than the general ones; for if in the course of analysis we find unsatis-

factory results with the latter, we can detect adulterations with certainty by the former.

Examination for Peanut Oil.—The density is almost the same. The sulphuric saponification, and above all the Cailletet process give us good results, but only the presence of arachidic acid $C_{22}H_{44}O_2$, melting point 75°, allows the sure recognition of the presence of peanut oil. We have adopted with slight modifications the method of Renard. Twenty grams of the oil are saponified by 20 cc. of a caustic soda solution 36° B., diluted in 100 cc. alcohol ninety per cent. The soap formed is precipitated by a fifty per cent. solution in alcohol of lead acetate, which must be neutral. After complete precipitation decant while warm and wash the residue with alcohol, which after being ground in a mortar is agitated with 200 cc. of ether. This operation is repeated three times to remove the last traces of lead oleate, soluble in ether. The residue is then put in a porcelain dish containing two or three liters of distilled water and fifty cc. hydrochloric acid. When decomposition is completed the solution is decanted and the fatty acids washed with distilled water, after which they are dried in an oven to remove the last traces of water, when they are dissolved in forty cc. of ninety per cent. alcohol. A drop of hydrochloric acid is added, and the mixture chilled to 15°. Peanut oil gives a generous deposition of arachidic acid crystals. These are washed twice, using twenty cc. each time of ninety per cent. alcohol, then three times with twenty cc. each time of seventy per cent. alcohol, in which arachidic acid is completely insoluble. The washing is complete when a few drops give no residue on evaporation. The acids are warmed slightly and treated with boiling absolute alcohol. After filtration the alcohol is evaporated in the oven at 100° till the weight of the residue remains constant. If the melting point of the residue is between 73° and 75° we can affirm the presence of peanut oil. We must be sure of the freedom of the fatty acids from all traces of oleic acid, which prevents their crystallization. This elaborate but truly scientific process must be carried out only by skilled hands.

Examination for Sesame Oil.—To recognize sesame oil in olive oil we can use the specific gravity, the sulphuric saponifi-

cation and Cailletet process, but especially the method which we use in our laboratory, and which consists of the reaction of hydrochloric acid and sugar upon the fat.

We do not operate on the glyceride, but on the derived fatty acids. If the oil is operated on directly we can obtain a red or pink coloration with a perfectly pure olive oil. I have frequently observed this coloration in my laboratory with olive oils from Tunis, Algiers, Molfetta, Bitonto (Italy), and more rarely with those from Provence. This coloration comes from the coloring matter dissolved in the juice which flows out along with the oil from the presses. This can be shown by treating the separated juice with hydrochloric acid and sugar with which it gives a coloration exactly resembling that given by sesame oil. It is, therefore, of the highest importance to work with pure fatty acids according to the process I am about to describe and which has been called the Milliau process.

Method of Operating.—We saponify fifteen cc. of the fat under examination with ten cc. of the solution of caustic soda, 36° B., with the addition of ten cc. ninety-two per cent. alcohol. When the boiling mixture becomes clear we add 200 cc. hot distilled water and boil to expel the alcohol. Then decompose with ten per cent. sulphuric acid. The fatty acids are removed from the surface in the pasty state, washed by shaking in a test tube with cold distilled water, after which they are heated in an oven to 105°. When the greater part of the water is eliminated and they commence to melt, we pour them on half their volume of pure hydrochloric acid which has been saturated in the cold with finely pulverized sugar. The mixture is shaken violently in the test tube. The presence of sesame is always distinctly indicated by the rose or red coloration of the acid solution. Other oils leave the acid colorless or communicate to it a slightly yellowish tinge. This reaction is extremely delicate and permits the sure recognition of the presence of one per cent. of sesame oil, not only in olive oil, but in all fatty mixtures as well as in soap.

This process was presented before the Academy of Sciences by M. Debray, February 20, 1888, and was awarded the gold medal by the Société d'Encouragement (Report of M. Muntz in the name of the Agricultural Committee, February 20, 1889).

Examination for Cotton-Seed Oil.—Till recently the detection of cotton seed in olive oil was considered impossible, and accordingly the different scientific societies and several chambers of commerce promised great prizes to the inventor of a process which would detect this adulterant. Two simultaneous methods were proposed, one by an Italian savant, M. Bechi, the other by myself. These two processes, which at first sight appear quite analogous and based upon the same reaction, differ completely from the scientific standpoint as well as in the exactness of the results obtained. M. Bechi has based his process upon the direct action of silver nitrate upon the oils. As long ago as 1878 we made some experiments with M. Puget of a similar nature, and the uncertainty of the reactions led us to abandon them entirely. M. Bechi's process possesses the serious inconvenience of causing us at times to reject, as adulterated, oils which are absolutely pure, and thus work injustice and create a considerable prejudice against the firms which have sold them. From results obtained at L'Institut National Agronomique de Paris as well as in other laboratories a deep coloration has been found with oils absolutely pure. Outside of these inconveniences, which are sufficient to cause us to throw it aside, it has the defect of being based upon a coloration having no well-defined chemical character, and it produces effects with different substances the action of which it is impossible to explain. The use of colza oil has the serious fault of bringing into the reaction a second oil which may be impure and completely nullify the results. Finally, the conclusions reached by the Italian scientific commission instituted at Rome by act of the ministry to study the Bechi process show that it is not certain on quantities of less than fifteen per cent. It solves the problem of finding less than ten per cent. no better than the Cailletet process, which shows the presence of fifteen per cent. The same commission declared also that olive oil containing glycerine, free fatty acid, formic and acetic acids, does not give a sure reaction with the Bechi reagent. Since all olive oils contain free fatty acid from several tenths of one per cent. for virgin oil to 100 per cent. in some industrial oils, what conclusions can we arrive at?

The Bechi process will give, from fifteen per cent. up, a brown

coloration of variable intensity. But in oil containing cotton-seed oil, which is fresh and well-refined, the only sort used for edible purposes, we have a very weak reaction; while with an olive oil perfectly pure, but containing organic or mineral matters in suspension or solution which have an action upon the reagents, we would have a pronounced coloration. That is why I gave up these uncertain and variable results obtained in 1878 and studied the action of nitrate of silver, not upon the oil itself, but upon the products of saponification derived from it. The results obtained exceeded my expectations and have been published by the press as the Milliau process.

Milliau Process, Mode of Procedure.—In a porcelain dish of 250 cc. capacity fifteen cc. of the oil to be investigated is heated to about 110°; a mixture of ten cc. of a solution of caustic soda at 36° B., and ten cc. of alcohol at 90° B. is then slowly poured on the oil. When the mass boils it becomes clear and homogeneous; 150 cc. of hot distilled water is added and boiling continued to expel the alcohol. The fatty acids are displaced with ten per cent. sulphuric acid solution added in slight excess, and the pasty acids immediately collected with a small platinum spoon. They are washed by shaking several times in a test tube with an equal volume of cold distilled water, the drops of water are drained off and they are then poured into a tube 2.5 cm. in diameter and nine cm. long; fifteen cc. of alcohol, ninety-five per cent., and two cc. of three per cent. nitrate of silver solution are added. The tube is protected from the light in a water bath at 90° C. till about one-third of the alcohol is expelled, which is replaced by ten cc. distilled water. The heating is continued a few minutes and the coloration of the insoluble fatty acids is observed. The presence of cotton-seed oil in any proportion whatever causes a mirror-like precipitate of metallic silver, which blackens the fatty acid of the mixture.

The members of the agricultural committee, composed of the greatest savants of France, MM. Pasteur, Eug. Tisserand, Boitel, Chatin, Heuzé, Hardi, Rissler, Schloesing, Ronna, Lavalard, Müntz, Prillisux, Muret, Bon Chénard, Liebaut, after having tested the process on 128 samples from different provinces have had the kindness to confer upon me the gold

medal for this method of analysis, and I will take the liberty to present the conclusions of their secretary, M. Müntz, director of the laboratories of L'Institut National Agronomique: "The preliminary saponification applied to the examination of fatty matters constitutes a new method on which we can confidently rely, not only in the analysis of edible products, but besides of those used for industrial purposes, and also for soaps. It has for effect not only to give an extremely sensitive reaction, but it also eliminates accidental and secondary products which can mask the reaction and deprive it of precision. Feb. 1889."

It is of course well understood that the same processes can be used for the detection of peanut, sesame, and cotton-seed oils in all other fatty mixtures.

Peanut Oil.—Peanut oil apart from the arachidic acid which it contains presents no special characters.

Sesame Oil.—The purity of sesame oil may be demonstrated by shaking ten cc. of oil, first with five drops of sulphuric acid at 53° B., then with five drops of nitric acid at 28° B. The oil undergoes a progressive change of color, through various shades from light-green to red; the final red coloring matter obtained by this oxidation process turns yellow by the action of alkalies and returns to its original color with acids. This curious phenomenon does not take place with other oils, but is obtained sometimes when working on sesame oils slightly adulterated; besides the industrial oils from hot pressing may give, though pure, negative results. I have devised a rapid process, especially applicable for the recognition of castor oil in the industrial sesame oils.

Milliau Process.—For the rapid identification of castor oil and sesame ten grams of the oil are shaken up with four drops of sulphuric acid at 66° B., a drop of nitric acid at 40° is added and shaken violently; pure sesame oil blackens immediately, while that containing castor oil remains turbid yellow.

Cotton-seed Oil.—From a chemical standpoint we have nothing particular to say. From the industrial point of view we may state that this product becomes more useful every day, that its cheapness allows everybody to use it, and this fat material daily augments its important place in the markets of the world.

We will go rapidly over the drying oils, poppy-seed, linseed, walnut, cameline, over those of the cruciferae, colza, rape, and mustard, as well as the oils of the sweet almond, hazel nut, and castor.

I will take the opportunity, however, to mention a rapid process which I have found for detecting the presence of castor oil in other oils. It is well known that the oils in general are soluble in petroleum ether, while castor oil, which distinguishes itself by its solubility in alcohol is likewise remarkable for its insolubility in petroleum ether. Unfortunately this insolubility disappears at the ordinary temperature when castor oil is adulterated wlth a small proportion of a soluble oil.

It is only necessary to demonstrate its insolubility by shaking in a test tube one volume of the oil to be examined with two volumes of petroleum ether, and cooling the mixture to minus 16° C., at the end of a few minutes the mass coagulates and the oil separates if castor oil is present, while the liquid remains homogeneous if it is pure. This phenomenon is especially curious because it will be noted that the freezing point of castor oil unmixed with petroleum ether is much lower than the majority of other oils.

SOLID VEGETABLE OILS.

Cocoanut and Palm Kernel Oils.—The production of these oils, which is continually increasing on account of their great value for the manufacture of soap during the last few years, has been made the object of several adulterations. Independently of the general, chemical, and physical characters of these oils, as well as the sulphuric saponification which I have mentioned, we can employ with good results the determination of the saturation equivalent with normal caustic soda solution.

These oils, containing a greater amount of the lower fatty acids, require, for their saturation, a larger quantity of sodium oxide. It is only necessary to determine the number of cc. of normal solution required for the saturation of five grams of the pure fatty acid to recognize these oils,—cocoanut oil 24.1, palm kernel oil 22.5, while the average of the other liquids is seventeen or eighteen cc. only. This process presents some inconveniences, especially when trying to discover small quantities of

adulteration, because it must be noted that the adulteration of these oils is only practiced within certain limits, otherwise their appearance would lead to its detection. Besides, when an oil is made from old or spoiled seeds the saturation is a little less.

Since the detection of adulterations in these oils depends on variations of a few tenths of a centimeter which might be caused by the variation of the oils themselves, or the method of operating, it is necessary to be very circumspect in the use of this truly scientific process.

With palm kernel oil this error might be greater because it is customary to balance the low saturation of the seed oils by the addition of cocoanut oil. As, for example, five grams of fatty acids from a mixture containing forty-five per cent. of palm kernel oil, forty-five per cent. cocoanut oil, and ten per cent. peanut oil, require precisely 22.5 cc. normal solution for their saturation.

Iodine Number.—This determination is very useful because these oils being very rich in saturated acids absorb much smaller quantities of iodine than most other oils. For example, peanut gives ninety-seven while cocoanut and palm kernel show only nine to sixteen. Unfortunately this difference of seven between the maximum and minimum throws us out of the way for small quantities of admixture, so much so that different authors fail to agree upon the average variation—some giving eight to nine only, while we are accustomed to find from thirteen to fifteen.

To resume this process, which, if it gave constant results, would be excellent, fails to give sufficiently accurate indications to detect adulteration of cocoanut oil when in small proportions. It is worse with palm kernel oil and of no value to distinguish between the two.

Continuing our investigations in another direction we have fortunately found a process which gives constant results when used upon these oils after a preliminary neutralization. We have discovered that cocoanut and palm kernel oils are entirely soluble in absolute alcohol. At a temperature of 30°–31° C. the former requires two volumes and the latter four volumes for complete solution.

It is a curious phenomenon that the smallest addition of vegetable or animal oils destroys this solubility in the same quantities of absolute alcohol. The solubility of the mixture is not proportional to its composition, but the mixture acts entirely like a distinct body.

Mode of Procedure.—First operation: Twenty cc. of the oil are shaken in a test tube with forty cc. of ninety-five per cent. alcohol. This indispensable preliminary treatment may give certain indications. Oil soluble in ninety-five per cent. alcohol, castor, resin oil, etc., are thus discovered, while Mowrah and Karité oils give a milky turbidity to the alcoholic stratum.

Second operation: Five cc. of the neutralized cocoanut oil are measured with a pipette into a graduated test tube, and ten cc. absolute alcohol added. The temperature is raised to 31°, the tube shaken violently for half a minute and then immersed in a water bath, kept at a temperature slightly above that of the tube. Pure cocoanut oil dissolves completely, and the solution remains clear. Any addition of another fatty matter causes precipitation. The material in solution being in a state of molecular equilibrium which is destroyed by the slightest modification.

Cocoanut oil, containing palm kernel oil, precipitates when the proportion of the mixture amounts to twenty per cent.; below this the mass remains turbid.

The verification of palm kernel oil is made in the same manner, only using twenty cc. absolute alcohol instead of ten, temperature remaining the same, 30–31°; five cc. of palm kernel oil, containing twenty per cent. of cocoanut oil, and above, dissolves in fifteen cc. absolute alcohol. In the same proportions pure oil does not dissolve and the mixture remains turbid.

A mixture of cocoanut, palm kernel, and peanut oil in such proportions that the oil would appear pure by the indices of saturation would be easily discovered by this process.

If we work at a lower temperature the proportion of absolute alcohol must be increased. For example, at 25–26° it is necessary to double the quantity; for five cc. of cocoanut oil, twenty cc. of alcohol, and for the palm kernel nut forty cc.

The same method may be used to determine the purity of co-

coanut and palm kernel cakes by first extracting the oil by means of a solvent and then operating on it in the manner described.

This process, which I have had the honor to describe, carefully executed will allow us to determine in a few minutes adulterations of these oils which might cause bad results in soap making, which employs hundreds of millions of kilograms per annum, and in agriculture which uses the cakes for cattle food. This process was presented to the Academy of Sciences, Oct. 10, 1892, by M. L. Troost, and has been the object of a favorable report to the minister of agriculture.

Let us pass rapidly over palm oil whose principal chemical properties possess nothing in particular except its partial solubility in absolute alcohol, which is ten per cent. Also, we will pass over the Mowrah, which, as I have already mentioned, is identified by the milky white turbidity which it communicates to alcohol by shaking.

Illepe oil, whose titer is 52.5°, and cotton-seed margarine or stearine, as it is called here, whose chemical reactions are the same as the oil itself, will not long detain us.

I wish to mention, while closing the description of the solid vegetable oils, Karité oil, which comes from the French province, Soudan, which I have been especially directed to investigate by the Chamber of Commerce, of Paris, and the Chamber of the Syndicate for the manufacture of soap and candles.

It was very important for us to know how to utilize the great forest which covered the largest part of French Soudan. It is composed chiefly of the wild acacia and the Karité or butter tree. The natives collect the nuts which fall from the trees, crush the kernels in a mortar, treat the pasty mass with boiling water, and skim off the fat which comes to the surface. This fat, which is solid at the ordinary temperature, is used as an edible butter.

Without speaking about the great uses to civilization of this product, I must mention its remarkable chemical properties. The fatty acids having a titer of 52.5° C. combine with soda to produce an extremely hard soap. For candle making they would probably give better results after preliminary treatment

to remove the resinoid matters which they contain and which hinder the crystallization of the fatty acids.

The presence of these resins lower the saturation to 14.9, and like the Mowrah this oil causes turbidity by shaking with alcohol (communication of M. Milliau to the Société d'Encouragement de Paris in 1892).

SOLID ANIMAL FATS, COLORED BROWN BY A STREAM OF CHLORINE GAS.

Butter.—The analysis of this material, which has attracted so much attention during the last few years, is not so difficult as some folks imagine, but requires to be carried on by the skill of a practical and experienced analyst. The specific gravity of butter is notably lower than that of tallow and lard. It is the same with the iodine number and the freezing point of the neutral fat. The saponification and the solubility in absolute alcohol on the contrary are notably higher.

We can combine with these different characters microscopical examination, melted butters showing under the microscope a collection of small regular spheres. Adulterated butter shows on the contrary abnormal figures as well as crystals which appear brilliant in the dark field of the polarizer.

By determining the fixed and volatile fatty acids and the solubility in alcoholic toluene, a skilled analyst familiar with these processes can easily determine admixtures above ten per cent. Below this point I do not believe any adulteration would be profitable. Natural butter contains about eleven per cent. soluble and volatile fatty acids, and eighty-seven per cent. fixed fatty acids. Besides alcoholic toluene dissolves it almost entirely.

Tallow.—The properties of tallow are so well known that they require no description. The distinction between beef and mutton tallow is a matter of interest, and presents great difficulties from the chemical standpoint, and we can only observe differences by taking the melting and solidifying points of the neutral fat and those of the fatty acids.

Lard.—Lard adulterated with cotton-seed oil is easily tested with nitrate of silver.

If the lard has been altered by time or other causes, it is necessary before using this reagent upon the fatty acids to take the precaution of purifying the fat in the manner in which I have indicated at the beginning of this article. We will thus avoid the slight reduction which is produced by decomposition products.

Some authors strongly believe in the use of the iodine number, which will give good results in the cases of large admixtures, as well as for quantitative analysis of such a mixture. But it is quite illusory for the detection of a small proportion of cotton-seed oil, for we must consider that the iodine number of pure lard varies within considerable limits, and it would be possible to correct for the high iodine number of cotton-seed oil by the addition of tallow, which will lower it appreciably. In such a mixture the action of nitrate of silver on the fatty acids allows us to discover adulteration even below five per cent. Tallow is detected by observing the crystallization of the fat from ethereal solution under the microscope.

The following oils, which frequently are used for the adulteration of agricultural fatty materials, are easily detected:

Fish Oil.—By its iodine number, its solubility in absolute alcohol, the presence of cholesterine, and finally the brown coloration by chlorine and red by caustic soda and phosphoric acid.

Resins.—By their density, their saturation, their solubility in absolute alcohol, and the deviation of the plane of polarization. Besides, it is easy to isolate them because the majority of compounds which they form with metallic salts are soluble in ether, while those of the fatty acids are insoluble.

Resin Oils.—By their property of not saponifying with caustic soda as well as by their insolubility in glacial acetic acid, and the characteristic purple coloration which they give with fuming stannic chloride.

Oleine Oil of Commerce.—By its solubility in alcohol and by its specific gravity.

Finally, *the mineral oils*, by their iodine numbers, their indifference to the action of caustic soda, and their insolubility in absolute alcohol at 15° C.

The processes which I have just enumerated are applicable

indiscriminately to fatty matters both edible and industrial, for the recognition of their purity with an approximation sufficient for the majority of cases.

The results obtained will be more conclusive if they are compared with those from products of the same origin and known purity.

Although the majority of the processes which I have just indicated were devised and perfected in my laboratory and have been adopted by several governments, I have not, however, the presumption to feel that we have definitely finished this question, but consider on the contrary that in the vast unexplored region of the fatty series we have only taken a few steps. But I feel at the same time that we are now in the possession of sensitive and scientific methods which permit us to detect mixtures, the determination of which has appeared impossible up to the last few years.

The mission with which I have been charged by the French and Tunisian governments at the demands of commerce and industry shows you to what extent these countries are desirous of facilitating the exportation of pure and satisfactory products. France furnishes the best growth of virgin olive oil from Aix en Provence and Nice. On its side Tunis furnishes as well, but at a less price, olive oils of excellent quality which we can certify without analysis, because the government imposes such heavy export and import duties upon seed and seed oils that it would be impossible to practice the smallest amount of sophistication.

Often people who deal in olive oils make the remark that pure oil is too strong and it is improved in flavor by a mixture with a seed oil. Without discussing this inexact statement I believe that it would be preferable in every instance for the dealer to buy pure olive oil and to make his own mixtures. He would thus profit by the difference in price, which averages about sixty francs per 100 kilograms.

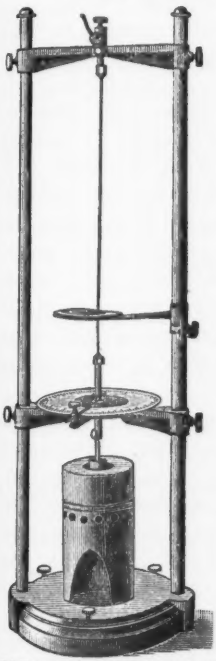
THE TORSION VISCOSIMETER.[1]

By O. S. Doolittle.

THE viscosity of an oil is recognized to-day by both the producer and consumer as the most valuable measure of its lubricating power, and yet we find no uniformity whatever in the manner of determining this essential property.

There are numerous vicosimeters in use, but no one of them has commended itself to the trade sufficiently to be adopted as a standard.

Many of them have their good features, but all have objections which have prevented any one from being accepted as standard and universally used.



DOOLITTLE'S TORSION VICOSIMETER.

The essentials of a good viscosimeter are:

(1) Accuracy, including both the ability to duplicate results obtained with an oil on the same instrument, and also on different instruments of the same make.

(2) Ease and rapidity of cleaning and manipulating, and the reducing of personal error to a minimum.

(3) Adaptability of a single instrument to all kinds of oil at all desirable temperatures.

The great majority of viscosimeters are built on the principle of allowing the oil to flow through an orifice and counting the number of seconds required for a certain quantity to flow out. Instruments constructed on this plan cannot be made to conform satisfactorily to all the above requirements.

As a rule it takes more time to clean and get the viscosimeters of this class ready for a test, than for the test itself. If the instrument is made with great care good duplicate results can be obtained with a perfectly clean oil,

[1] *Drugs, Oils and Paints*, June, 1893.

but if by any chance a slight particle of dirt gets into the oil the orifice is liable to become obstructed and the results vitiated.

At the same time these results are but comparative, and poorly comparative at the best, as the head of oil is constantly changing from the moment the flow begins, and the rapidity of the flow must necessarily depend more or less upon the specific gravity of the oil.

That the personal error is a large one will hardly be disputed by any who have worked with these instruments. Again, you will find as a rule several different viscosimeters with varying orifices required for the different oils, a small hole being the best for light oils and a large one for the heavy products.

Thus we have no regular scale of viscosities ranging upward from some recognized zero point, which by the way water furnishes admirably.

This is a most unfortunate state of affairs, as it deprives us of a standard by which the value of all oils could be readily recognized and intelligently understood. When we speak of a temperature of 212° it is well understood because the Fahrenheit thermometer is recognized as a standard in this country, but when we refer to a viscosity of 125 seconds it has no meaning whatever unless it is accompanied by the name of the instrument on which it was obtained, and oftentimes the name of the observer as well, thus necessitating the carrying in mind of the ratio of numerous instruments to each other, a very perplexing and unsatisfactory task.

Having experimented with a number of these viscosimeters in the laboratory of the Philadelphia and Reading railroad, we found them so very unsatisfactory where rapid and accurate work is required that we abandoned them all and designed an instrument on an old principle which has been used in measuring almost everything from a delicate electrical current to the quantity of cream in milk, an operation at times requiring a still more sensitive instrument, *i. e.*, that of the torsion balance.

The principle has not failed us in this case, and in the torsion viscosimeter we have an instrument which, during the year we have had it in daily use, has proved itself reliable, accurate, and satisfactory in every way.

It is very easy to clean and manipulate, is adapted to oils of all ranges of viscosity from kerosene up, and reduces the personal error to a minimum.

A glance at the cut will show how the principle has been applied.

A steel wire is suspended from a firm support and fastened to a stem which passes through a graduated horizontal disk, thus allowing us to measure accurately the torsion of the wire. The disk is adjusted so that the index point reads exactly zero, thus showing that there is no torsion in the wire.

A cylinder two inches long by one and a half inches in diameter, having a slender stem by which to suspend it, is then immersed in the oil and fastened by a thumb-screw on the lower part of the stem to the disk. The oil is surrounded by a bath of water or paraffine wax according to the temperature at which it is desired to take the viscosity. This temperature being obtained, while the disk is resting on its supports, the wire is twisted 360° by means of the knob at the top. The disk being released, the cylinder rotates in the oil by virtue of the torsion of the wire.

The action now observed is identical with that of the pendulum.

If there was no resistance to be overcome, the disk would revolve back to zero, and the momentum thus acquired would carry it to 360° in the opposite direction. What we find is that the resistance of the oil to the rotation of the cylinder causes the revolution to fall short of 360°, and that the greater the viscosity of the oil the greater will be the resistance and hence the retardation. We find this retardation to be a very delicate measure of the viscosity of an oil.

There are a number of ways in which this viscosity may be expressed, but the simplest we have found to be directly in the number of degrees of retardation between the first and second complete arcs covered by our pendulum. For example, suppose we twist the wire 360° and release the disk so that rotation begins. In order to obtain an absolute reading to start from, which shall be independent of any slight error in adjustment, we ignore the fact that we have started from 360°, and

take as our first reading the end of the first swing. Suppose our readings are as follows:

Right, 350; left, 338; right, 328, and keeping in mind the vibrations of the simple pendulum we will see at once that we have read two complete arcs whose difference is 22° computed as follows:

1st arc, Right 350° + Left 338° = 688°
2d arc, Left 338° + Right 328° = 666°

22° retardation

In order to secure freedom from error we take two tests—one by rotating the wire to the right, and the second to the left. If the instrument is in exact adjustment these two results will be the same, but if it is slightly out, the mean of the two readings will be the correct reading.

It will also be noticed that if the exact retardation due to the oil alone is to be obtained we must subtract the factor for the resistance due to the air and the wire itself. These are readily obtained by allowing the cylinder to rotate in the air and determining the retardation exactly as we have done above. This factor remains constant for each instrument and is simply deducted from all results obtained.

The torsion viscosimeter is free from many of the objections noticed in other instruments, and has given entire satisfaction during the period it has been in use. It is independent of the gravity of the oil and of any reasonable amount of dirt which may get into it. It is applicable to all grades of oil regardless of their character or fluidity. The viscosity of an oil can be taken at any temperature as many times as may be desired without any inconvenience from being obliged to handle the hot oil.

This I think is an important point, as the practice in common use of determining the viscosity of a cylinder stock at 212° Fahrenheit does not tell us what we want to know. We should know the viscosity of an oil at the temperature at which it is to be used, which, in the case of cylinder stock, is in the neighborhood of 350°. I have repeatedly found oils tested at 212° simply reversing their comparative values when heated to 350°.

We need a viscosimeter with which the viscosity can readily

be determined at a high temperature with a minimum amount of trouble. By means of a paraffine bath for our oil we have no trouble whatever in doing this with the torsion viscosimeter.

When one oil has been tested sufficiently the cylinder can be taken out in a moment, wiped off, and is then ready for another oil.

The necessity of a standard instrument which shall be recognized by the trade as such cannot be too forcibly emphasized, as the present state of affairs is most annoying to both producer and consumer, leading as it so often does to misunderstanding and financial loss.

The torsion viscosimeter is the standard instrument of the Philadelphia and Reading railroad, but this simply means that it is the best we know of at present. We are, however, always open to conviction, and if any better method of determining the viscosity of an oil is invented we will be very glad to adopt it.

FIFTH GENERAL MEETING.

THE following resolution was adopted by the Council of the American Chemical Society, June 7, 1893:

Resolved, That the General Meeting of the American Chemical Society for the summer of 1893 be held in Chicago at such date as may be determined by the committee appointed by the society to co-operate with the World's Fair Auxiliary of the Columbian Exposition in arranging for an International Congress of Chemists.

In accordance with the terms of this resolution the committee to co-operate with the World's Fair Auxiliary have determined that the Fifth General Meeting of the Society shall be held during a period beginning August 21, 1893, in conjunction with the World's Congress of Chemists to be held under the auspices of the World's Fair Auxiliary and the American Chemical Society.

The committee desire to call to the attention of the members of the society and of American chemists generally, the fact that in view of the large number of eminent chemists, home and foreign, who have already signified an intention to attend the World's Congress of Chemists, and the valuable character of

the papers that will be presented, this meeting will doubtless be the most important gathering ever held by chemists in the United States. The time and place are therefore particularly auspicious, and the promise of a most successful and enjoyable occasion very great. The meetings will be open to all chemists in good standing, and in order that a program of proceedings may be arranged in advance, it is earnestly requested that those who desire to offer papers will forward titles thereof as early as August 1, to Prof. Wm. McMurtrie, 106 Wall street, New York City, care of New York Tartar Co.

It is also the hope of the committee that in view of the important part taken by the American Chemical Society in calling the World's Congress of Chemists, the society will be represented by a large number of its members in this meeting. This is due to the foreign chemists who have accepted the invitations extended by the society.

In matters of transportation to, and quarters in, Chicago, members of the society will have all the advantages provided for those who will attend the World's Congress of Chemists. Those desiring information with this regard may obtain it by addressing Prof. John H. Long, No. 2421 Dearborn avenue, Chicago, Ill.

It is requested that as soon as possible after their arrival in Chicago, members will report at the Congress Headquarters in the Art Institute Building, Lake Front and Adams street, and register their names and Chicago addresses so that they may receive without delay such notices and information as the committees may have to distribute at that time.

WM. McMURTRIE,
C. F. CHANDLER,
A. B. PRESCOTT,
C. E. MUNROE,
H. W. WILEY,
Committee to Co-operate with the World's Fair Auxiliary.

PATENTS OF INTEREST TO CHEMISTS.

EDITED BY ALBERT H. WELLES.

Ore Separators, etc.—James B. Montgomery has a new form of ore concentrator (495,003). Orrin B. Peck adds one more to his list of patents on a centrifugal ore separator (495,681), and M. W. Iles covers his process of and apparatus for the extraction of matte from slag by numbers 494,570 and 494,571. A. L. Engelbach and S. E. Bretherton have an apparatus for reducing and smelting sulphide ores (496,250). Charles M. Allen takes out three related patents: 496,032 treats of his process of smelting ores and refining metals, which is conducted in a converter (496,033), which has a patent tuyere (496,034). The ores are fed into this converter and the blast is passed through the molten metal for a time, then checked by plugging the tuyeres to permit slag, matte, and metal to separate in the converter at different heights, and enough molten metal is retained each time to ignite the next charge. Thomas A. Edison has a roller for crushing ores (498,385). N. L. Raber (497,603) and A. B. Kittson (497,669) have each an amalgamator, and Uriah Cummings patents his ore crusher (498,424). C. B. Walker has a new form of ore concentrator (497,843), and Charles G. Brown a patent ore tank for leaching (497,856). Orrin B. Peck protects his centrifugal ore separator by 497,204, while F. H. Wheelan (498,597), W. P. Miller (497,474), Charles G. Buchanan (497,117), and Albert M. Bair (496,391) have each a separator. William Stubblebine's metallurgical furnace (498,089) and M. Mannaberg's steel-smelting furnace (498,670) are recent issues, as well as Joseph McClelland's tuyere (498,565). An apparatus for removing matte from slag is the joint invention of Thomas Drohan and Thomas Pearce (496,823). John W. Marshall is the patentee of an ore-stamping mill (493,384) and of an ore stamp (493,385). Charles J. Fauvel has a new furnace for the treatment of refractory ores (493,076). C. W. Beehler is the inventor of a hydrothermal mining process (497,513), which consists in filling a closed casing with a liquid body, inserting in bore hole, and heating by electric resistance coil, so that the expansive force of the liquid may be exerted within the drill hole.

Lead.—"Sublimed lead pigments" are prepared by E. O. Bartlett by driving off lead fumes from lead-bearing material in suitable furnaces, carrying the fumes through flues at a red heat, and separating the purified fumes by screening from the gaseous products (496,038). Two more patent processes for the manufacture of white lead. 496,109, A. B. Browne, patentee, consists in placing lead in a solution of a nitrate of an alkaline base; the current is then passed and the alkaline hydrate formed at one pole precipitates the lead which has been dissolved, and the resultant lead hydrate is dried in an atmosphere of carbon dioxide. 495,490 is the invention of Andrew Honman and Victor Vulliez. Lead sulphate is dissolved in a solution of caustic soda or potash or ammonium acetate, heated, and precipitated with an alkaline carbonate and dried.

Fertilizers.—Ludwig Rissmüller manufactures superphosphates from kettle residues of glue factories by mixing residues with warm sulphuric acid (50° B.) and heating to 200° F. After the nitrogenous matters are dissolved by the acid, the gypsum is precipitated and the fat rises to the surface, the separated solution is treated with phosphate of lime to take up all the sulphuric acid (494,939). 494,940 is a similar process, applied to animal carcasses, etc. Omar T. Joslin treats tank water with sulphuric acid, adds five to thirteen per cent. of a concentrated solution of waste fullers' earth dissolved in sulphuric acid, and dries the product at 300°–350° F. (495,042); and 495,043 is also his patent for manufacturing fertilizer from tank water in which acid phosphate of calcium in combination with fullers' earth is used, instead of the latter alone. Philip C. Hoffman treats pulverized phosphates of the "Florida phosphate" class with sulphuric acid, heats, and maintains the temperature above the normal temperature occasioned by chemical action, which is usually 50° C., but yet below that at which pyrophosphates are formed (496,687). Robert Reiman chemically dissolves natural bone or bone meal into its constituents, precipitating these elements, filters and washes and afterwards mixes with albumen, aluminum sulphate, and cellulose in solution, dries partly, then subjects to a high temperature and powerful pressure simultaneously, thus manufacturing artificial bone (494,891).

Gold and Silver.—Wm. Birkin subjects ores to the action of potassium cyanide, potassium ferricyanide, and hydrogen peroxide to extract the precious metals (494,054). For the lixiviation of gold and silver ores Samuel R. Whitall first mixes them with either caustic soda or potash, then treats with a solution of potassium cyanide and sodium hyposulphite and precipitates the precious metals by electrolysis (495,715). Fred. Webb has an apparatus for extracting precious metals from their ores (495,385), and John F. Wiswell treats ores as follows: Mercury is submerged in a solution of common salt which is electrolyzed. The calomel formed is dissolved in aqua regia, forming mercuric chloride, while the undecomposed salt solution is further electrolyzed, forming sodium hypochlorite. The ores are then treated simultaneously with both solution of the hypochlorite and mercuric chloride, and the dissolved metals are precipitated by adding bright iron fragments (495,212). F. W. Cleghorn separates precious metals from their ores by filtering through the ores a solution of sulphuric acid and sodium chloride, placing metallic iron in the filtrate and passing the current (497,014), while Henry Parkes uses one of two methods to accomplish the same end, treating the ores with potassium cyanide in the presence of oxygen under pressure (496,950), or treating with chlorine and then agitating with oxygen under pressure (496,951). Jabez Turton proposes to separate metals from ores by treating the ore or mineral to the action of a nitrate of an alkali metal, sodium chloride, and sulphuric acid, separating the earthy matters and precipitating the metals (494,044).